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Edwards AFB, Ca 93523

FIFTH PROGRESS REPORT

STORABILITY INVESTIGATIONS OF WATER STORAGE EVALUATION AFTER SEVEN YEARS

Aerojet Liquid Rocket Company
Sacramento, California

E. M. VANDER WALL
G. R. JANSER

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FOREWORD

This report covers the work performed under Contract F04611-72-C-0062, "Storability Investigations of Water," performed by the Aerojet Liquid Rocket Company at Sacramento, California, and conducted under Air Force Project Task 305911 VD. The performance period covered from 1 October 1979 to 30 September 1980.

The program manager is R. L. Friedman; the project manager is Dr. E.M. Vander Wall. The experimental work was conducted by Dr. Vander Wall; R. L. Beegle, Jr., senior chemist; J. A. Cabeal, senior chemist and G. R. Janser, metallurgy specialist.

The program was administered under the direction of the Air Force Rocket Propulsion Laboratory, Mr. Herman Martens, Project Engineer.

This report has been reviewed and is approved for release in accordance with the distribution statement on the cover and on the DD Form 1473.

Herman N Martens
Herman Martens
Project Engineer

Forrest S. Forbes
Forrest S. Forbes, Chief
Propellant Systems Section

For the Commander

Edward E. Stein
Edward E. Stein, Deputy Chief
Liquid Rocket Division

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20. ABSTRACT (Continue on reverse side if necessary and identify by block number)

The objective of this program is to gather data that will permit the Air Force to assess the long-term storage characteristics of water, particularly with regard to formation of particulate matter, so that the feasibility of long-term storage of water for use as a transpiration coolant can be determined. Five metallic materials of construction are included in the program: 304 stainless steel; A-286 (aged) steel, 17-4PH (aged) stainless steel;

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Abstract (cont)

Inconel 718 (aged); 6Al-4V titanium (STA). Two types of water are used in the program: 1) oxygen-saturated, deionized, filtered, and 2) oxygen-free, deionized, filtered. Using the filtered, deionized waters, ten-year storage tests have been initiated in 304 and 17-4PH stainless steel, A-286 (aged) steel, Inconel 718 (aged) and 6Al-4V titanium (STA) containers.

Evaluation of water and containers stored for seven years has been completed. The data show that both oxygen-saturated and oxygen-free water can be stored in appropriate metal containers for the selected time periods without detrimental particulate matter formation or significant changes in the quality of the water. It is in excellent condition for transpiration coolant purposes.

TABLE OF CONTENTS

TABLE LIST

LIST OF FIGURES

SECTION I

INTRODUCTION

Inherent in the concept of transpiration cooling is the requirement that the coolant remain free of particulate matter which may clog the passages of the cooling surface. The object of the "Storability Investigations of Water" program is to gather data that will permit the Air Force to assess the long-term storage characteristics of water, particularly with regard to formation of particulate matter. This report is the fifth progress report to document the experimental results from the eighty-four month storage tests of water in selected metal containers for the ten-year program conducted under Contract F04611-72-C-0062.

The five metallic materials used for container material in the program are:

- 304 stainless steel
- A-286 (aged) steel
- 17-4PH (aged) stainless steel
- Inconel 718 (aged)
- 6Al-4V Titanium (STA)

Two types of water are considered in the program:

- oxygen-saturated, deionized, filtered water and
- oxygen-free, deionized, filtered water

Three categories of tests are used for obtaining the data necessary for assessment of the storability of water. They are:

- Water Characterization
- Biological Characterization
- Container Examination

The investigations which led to the selection of the candidate metallic materials for tankage for use in the ten-year storage of waters are reported in AFRPL-TR-73-94 "STORABILITY INVESTIGATIONS OF WATER," VOLUME I: EXPERIMENTAL STUDIES, FINAL REPORT, Aerojet Liquid Rocket Company, Sacramento, California, December 1973. The results from the first year of storage are reported in AFRPL-TR-74-76 "STORABILITY INVESTIGATIONS OF WATER, LONG TERM STORAGE EVALUATION," FIRST ANNUAL REPORT, Aerojet Liquid Rocket Company, Sacramento, California, December 1974. The results from the second year of storage are reported in AFRPL-TR-75-62 "STORABILITY INVESTIGATIONS OF WATER, LONG TERM STORAGE EVALUATION," SECOND ANNUAL REPORT, Aerojet Liquid Rocket Company, Sacramento, California, December 1975. The results from the third year of storage are reported in AFRPL-TR-76-95 "STORABILITY INVESTIGATIONS OF WATER, LONG TERM STORAGE EVALUATION," THIRD ANNUAL REPORT, Aerojet Liquid Rocket Company, Sacramento, California, December 1976. The results from the

Introduction (cont.)

fourth year of storage are reported in AFRPL-TR-77-74 "STORABILITY INVESTIGATIONS OF WATER, LONG-TERM STORAGE EVALUATION," FOURTH ANNUAL REPORT, Aerojet Liquid Rocket Company, Sacramento, California, December 1977.

This annual report is presented in three sections: (1) Introduction, (2) Experimental Results and Discussions, and (3) Conclusions and Recommendations. In addition, there are two appendices provided for the convenience of the reader: Appendix A, Fabrication and Treatment Procedures for Water Containers, which documents the history of the tanks, and Appendix B, Silting Index Measurement, which describes a clogging tendency test.

SECTION II

EXPERIMENTAL RESULTS AND DISCUSSIONS

BACKGROUND INFORMATION

The purpose of the long-term storage tests is to demonstrate that water can be stored without formation of significant quantities of particulate matter and with insignificant corrosion of appropriate metal containers for time periods of at least ten years in a controlled environment. The background information is presented for the convenience of the reader and documents the initial conditions of the selected containers and waters prior to the storage periods. The discussion is presented under the following headings: (1) Materials Selection, (2) Container Sterilization and Filling, and (3) Storage Conditions.

A. MATERIALS SELECTION

1. Waters

Based on the data derived from the preceding experimental work (Reference 1), it was apparent that both oxygen-saturated and oxygen-free water were acceptable candidates for long-term storage tests. Further, the filtration of the water through a 0.22 micron pore size absolute filter was demonstrated to remove microorganisms effectively. Thus, the water used to fill the containers was passed through an activated charcoal bed to remove organic compounds and through two mixed-bed ion exchangers to obtain water that had an electrical resistance value of 1 megohm/cm or greater. The water was transferred through 0.22 micron filters into a 5-gallon stainless steel supply tank. To ensure the saturation of the water with oxygen, filtered oxygen was purged through the water in the supply tank for a minimum of 15 minutes. To obtain oxygen-free water, the water in the supply tank was heated to the boiling point of water for one hour while being purged with filtered nitrogen obtained from the boil-off of liquid nitrogen. The tank was then pressurized with the filtered nitrogen, allowed to cool to ambient temperatures, and then repressurized with the filtered nitrogen. The outlet of the supply tank was fitted with a Twin 90 Filter* unit to assure the sterile characteristics of the water used to fill the storage containers.

2. Metals

The selection of the materials of construction for the long-term storage test containers was based on the results of the laboratory investigations (Reference 1). The aluminum alloys were eliminated from

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Ref. 1 E. M. Vander Wall, R. E. Anderson, G. R. Janser, Storability Investigations of Water, Volume I, Experimental Studies, AFRPL-TR-73-94, Contract F04611-72-C-0062 (December 1973).

* A 0.22 micron pore size, absolute filter pack available from Millipore Corporation, Bedford, MA.

consideration due to their introduction of insoluble corrosion products which are a source of particulate matter in the water. Because test results on the remainder of the seven candidate materials were not discriminatory, choice was made on the basis of selecting not more than one alloy from each class of material. The one class of material with more that one representative was the 18% chromium - 8% nickel austenitic stainless steels, i.e., 304L, 347 and Arde-form 301. Hence two of these materials were eliminated to provide the five materials required for container fabrication. The 304L stainless steel was selected due to its attractiveness as an expulsion bladder material. Hence, the selected materials are: 304L stainless steel, A-286 (aged), 17-4PH stainless steel, Inconnel 718 (aged), and 6Al-4V titanium (STA). During fabrication, some 304 stainless steel parts were incorporated into the 304L stainless steel containers, consequently identified as 304 stainless steel containers. The fabrication procedures, the heat treatment cycles, the cleaning procedures, and the passivation procedures to which the containers were subjected are presented in Appendix A of this report.

B. CONTAINER STERILIZATION AND FILLING

Following the final rinsing with filtered, deionized water and the subsequent drying of the containers in a vacuum chamber, the containers were wrapped with reusable sterilization paper. The wrapped containers were then sterilized in an autoclave at 250°F with 15 psig steam for 30 minutes, followed by a 30-minute drying period. The containers were then stored in the paper to maintain their sterile condition.

All the steps required to fill the containers with water were conducted in a sterile, laminar-flow bench. The tanks were removed from the wrapping paper in the laminar-flow bench. The tanks were weighed empty, then weighed when filled completely with sterile water to determine the total volume of the tank. The water was drained out and the tank was rinsed once more with the sterile water. A sample of the rinse water was checked for pH, conductivity, and Silting Index (see Appendix B). If the values indicated that particulate matter and dissolved species were not present, the tank was considered ready for filling; if the values indicated that contaminants were present, the tank was rinsed until there was no evidence of contamination. A Silting Index value of 1 or less for the rinse water when using the filter with a cross-sectional area of 1.0 mm^2 was used as the criterion that no significant quantity of loose particulate matter remained in the containers.

Before the final filling with oxygen-saturated deionized water, the tank was purged with oxygen from a filtered, supply. The tank was then filled with the water and a sample was withdrawn for pH conductivity, and Silting Index measurements. The ullage was adjusted to the ten percent value by weighing the container and its contents; the ullage space was purged with the filtered oxygen, and the container was capped with a sterile, tapered plug made from the same material as the container. The plug was seated in the fill-tube by use of a hammer. The containers were filled with the oxygen-free, deionized water in a analogous manner, except that filtered nitrogen instead of oxygen was used for purging and blanketing the container.

The final sealing of the containers was accomplished by GTA-welding the fill-tube/plug interface. The welds were inspected visually for any apparent anomalies. None were found. Then the containers were labeled for the long-term storage test and placed in plastic bags.

The sampling plan for the long-term storage tests is to 1) remove one container of each material with the two types of water for inspection and evaluation every six months for a period of four years, 2) evaluate one set of containers after seven years, and 3) evaluate the remaining set of containers after ten years. The contents will be characterized with respect to pH, conductivity, particulate content, and biological activity, and the containers themselves will be subjected to metallurgical examination if the other test data indicate that this is required.

C. STORAGE CONDITIONS

The storage area for the water containers is an air-conditioned room which is monitored continuously to document that the temperature is maintained at 70 ± 10°F and that the relative humidity is maintained at 50 ± 25 percent. The containers are stored in a closed metal cabinet to protect them from an accumulation of dust, and the containers themselves are covered with plastic bags to prevent direct contact with foreign metal surfaces. The containers are visually examined on a weekly basis.

WATER CHARACTERIZATION

After six-month intervals of storage at the conditions defined above, ten containers are removed for evaluation. They consist of two containers of each selected material, one containing oxygen-free water and the other containing oxygen-saturated water.

A. PROCEDURES

After the initial six-month storage period, the water containers were washed with deionized water and then placed in a sterile, laminar-flow bench for further handling to remove the stored water. The outlet of the container was rinsed repeatedly with filtered, deionized water to remove any contaminants and then briefly subjected to a torch flame to sterilize the exterior of the metal. In the subsequent six-month storage periods, the water containers were immersed in a 95% ethanol bath prior to placement in the sterile, laminar-flow bench. Subsequent to their removal from the bath and placement in the laminar-flow bench, the residual alcohol on the tank surface was

A. Procedures (cont.)

removed by burning. The outlet of the container was repeatedly exposed to a torch flame to assure a sterile condition. All the containers were opened in an identical manner. A sterile tubing cutter was used to sever the fill-tube. For the six-month, twelve-month, and eighteen-month storage periods, the water was expelled from the containers by inserting a sterile stainless steel capillary tube into the fill-tube of the container. Filtered, gaseous nitrogen was passed through the capillary tube while the container itself was kept inverted. The first several ml of water were used to flush the tube, then discarded. Subsequent samples of water were collected for measurement of pH, electrical conductivity, dissolved solids, particulate matter, flow behavior, and for characterization with regard to possible biological contamination. For the twenty-four month and subsequent storage periods, special cannula were fabricated so that the water could be expelled from the container while it was maintained in its normal upright position. The cyclic insertion and withdrawal of the cannula for the various samples was avoided by controlling the nitrogen flowrate used to pressurize the container during the water expulsion.

The measurement of the pH was made by using a standard pH meter with a calomel reference electrode and a glass indicator electrode. The electrical conductivity of the water was measured by using a Balsbaugh Conductivity Meter (Model No. 900-.01T) with a standard dip cell. The content of dissolved solids in the water was determined by evaporating 200-300 ml samples of the water to dryness and then weighing the residue. In addition, any particulate matter which collected on the 0.8μ filter of the flow behavior device was examined microscopically and sized. The flow behavior of the water was evaluated by using a Silting Index Apparatus (see Appendix B for description), which permits filtration of the liquid through a known area (1.0 mm^2) at a constant pressure to allow recording of the flow decay due to the presence of particulate matter as a function of time. The standard method of the test is described in ASTM F52-69. The data are reported as Silting Index values, i.e., the greater the value, the greater the degree of contamination by small particulate matter.

B. DISCUSSION OF RESULTS

The data obtained from the tests are presented in Tables 1 to 5. The data obtained during the initial loading of the containers, as well as the data for the preceding storage intervals, are included in the tabulation to facilitate comparison and identification of trends. The baseline data are labeled as initial, and the data obtained after the storage periods are labeled as final.

TABLE 1

DATA INDICATIVE OF THE STORABILITY OF WATER IN 304 STAINLESS STEEL CONTAINERS

Container Number	Exposure Period Months	Type of Water	pH Initial	pH Final	Resistance megohm/cm Initial	Resistance megohm/cm Final	Silting Index Initial	Silting Index Final	Total Solids Content, mg/l Initial	Total Solids Content, mg/l Final	Biological Activity Initial	Biological Activity Final	Particulate Matter Characteristics
3	6	O_2-free	7.0	7.2	1.75	0.84	0.23	1.06	<1	6	-	X	3.2 x 1.6μ platelets
11	6	O_2-sat.	7.0	7.2	1.75	0.88	0.90	1.20	<1	<1	-	X	8 x 8μ platelets
5	12	O_2-free	7.0	7.1	1.80	0.83	0.13	0.68	<1	<1	-	-	1 x 4μ platelets
4	12	O_2-sat.	7.0	7.3	1.75	0.97	0.47	2.35	<1	<1	-	X	Mat of 3μ particles
6	18	O_2-free	7.0	7.3	1.90	1.02	0.29	0.99	<1	4.5	-	-	4x4μ irregular particles
1	18	O_2-sat.	6.6	7.2	1.75	0.81	0.22	0.90	<1	27	-	+	4x4μ irregular particles
2	24	O_2-free	7.0	6.9	1.80	0.99	0.35	0.44	<1	6	-	-	48x24μ irregular platelet
7	24	O_2-sat.	6.9	7.1	1.70	0.93	0.05	0.73	<1	2.5	-	+	32x16μ irregular platelets
10	30	O_2-free	7.0	7.2	2.00	1.15	0.09	2.03	<1	3.0	-	+	Many particles 8x8μ, dark orange coloration
8	30	O_2-sat.	7.0	7.3	1.75	0.95	0.90	1.73	<1	<1	-	+	~2μ irregular particles
13	36	O_2-free	7.0	7.2	1.80	1.05	0.36	0.66	<1	2	-	-	Agglomerated 2-4μ particles
9	36	O_2-sat.	6.8	7.3	1.70	0.85	0.55	0.85	<1	7	-	+	~1μ irregular particles
14	42	O_2-free	7.0	7.1	1.85	0.95	0.30	1.60	<1	16	-	-	Many 1-2μ particles
12	42	O_2-sat.	6.8	6.8	1.80	0.73	0.60	0.75	<1	<1	-	+	Many 1-2μ particles
15	48	O_2-free	7.0	7.4	1.80	1.22	0.14	0.96	<1	11	-	-	Many 1-2μ particles
17	48	O_2-sat.	6.8	7.3	1.80	1.0	0.35	0.09	<1	<1	-	+	Mat of 1μ particles
16	84	O_2-free	7.0	7.0	2.00	1.20	0.02	0.73	<1	<1	-	+	Many ~1μ particles
18	84	O_2-sat.	7.0	7.4	1.70	0.81	0.56	0.33	<1	<1	-	+	Many ~1μ particles

TABLE 2

DATA INDICATIVE OF THE STORABILITY OF WATER IN A-286 CONTAINERS

Container Number	Exposure Period Months	Type of Water	Water Characterization: pH Initial	pH Final	Resistance megohm/cm Initial	Resistance megohm/cm Final	Silting Index Initial	Silting Index Final	Total Solids Content, mg/l Initial	Total Solids Content, mg/l Final	Biological Activity Initial	Biological Activity Final	Particulate Matter Characteristics
6	6	O_2-free	7.2	7.2	1.75	1.18	0	0.57	<1	<1	-	-	8 x 8μ platelets
13	6	O_2-sat.	7.0	7.2	1.80	0.97	0.54	1.11	<1	<1	-	X	3.6μ platelets
A-3	12	O_2-free	7.2	7.7	1.80	1.35	0	0.67	<1	<1	-	X	1 x 3μ platelets
19	12	O_2-sat.	6.9	6.7	1.80	1.08	0.46	0.65	<1	<1	-	-	3 x 3μ platelets
A-10F	18	O_2-free	7.2	6.4	1.75	1.04	0.00	0.06	<1	6	-	-	4x4μ irregular particles
A-2	18	O_2-sat.	7.0	6.8	1.80	1.17	0.73	0.24	<1	<1	-	+	8x4μ irregular particles
7	24	O_2-free	7.2	7.0	1.80	1.53	0.05	0.11	<1	4.5	-	+	7x7μ irregular platelets
4	24	O_2-sat.	6.7	7.1	1.80	1.26	0.43	0.16	<1	6.5	-	+	24x24μ Agglomerated particles
A-20F	30	O_2-free	7.2	7.3	1.80	1.16	~0	0.45	<1	<1	-	-	2μ Irregular particles
A-1	30	O_2-sat.	6.7	7.3	1.70	1.10	0.39	0.69	<1	<1	-	+	2μ Particles, very few observed
A-4	36	O_2-free	7.2	7.2	1.80	1.25	~0	0.35	<1	11.5	-	-	2μ Particles
10	36	O_2-sat.	6.7	7.2	1.80	1.37	0.98	0.40	<1	2	-	+	2μ Particles and agglomerates
A-5	42	O_2-free	7.0	7.1	1.95	1.05	0.04	0.86	<1	7	-	+	1 to 2μ particles and agglomerates
14	42	O_2-sat.	6.8	7.0	1.70	0.88	0.42	0.14	<1	<1	-	+	Many 1 to 2μ particles
8	48	O_2-free	7.0	7.2	1.80	1.80	0.41	0.21	<1	<1	-	+	Many 1 to 2μ particles
15	48	O_2-sat.	6.7	6.8	1.80	1.45	1.09	0.17	<1	4.5	-	+	Mat of ~1μ particles
16	84	O_2-free	7.0	6.9	1.90	1.20	0.57	0.78	<1	<1	-	-	Many ~1μ particles
17	84	O_2-sat.	7.0	7.0	1.60	1.80	1.02	0.42	<1	<1	-	+	1-2μ particles

TABLE 3

DATA INDICATIVE OF THE STORABILITY OF WATER IN 6Al-4V TITANIUM CONTAINERS

Container Number	Exposure Period Months	Type of Water	Water Characterization: pH Initial	pH Final	Resistance megohm/cm Initial	Resistance megohm/cm Final	Silting Index Initial	Silting Index Final	Total Solids Content, mg/l Initial	Total Solids Content, mg/l Final	Biological Activity Initial	Biological Activity Final	Particulate Matter Characteristics
2	6	O_2-free	7.0	7.4	1.90	0.84	0	0.69	<1	3.7	-	-	7.2 x 7.2μ + 5.4 x 5.4μ platelets
1	6	O_2-sat.	7.0	7.2	1.70	1.05	0.86	1.80	<1	1	-	-	3.6 x 3.6μ platelets
5	12	O_2-free	7.0	7.6	1.90	0.98	0.51	0.73	<1	<1	-	-	1 to 3μ particles, few 50μ agglomerates
3	12	O_2-sat.	7.0	7.5	1.65	0.82	0.10	1.17	<1	<1	-	-	1 to 2μ particles agglomerated as a mat
7	18	O_2-free	7.0	6.6	1.95	0.75	0.00	5.00	<1	<1	-	-	2x2μ Irregular particles
4	18	O_2-sat.	7.0	7.5	1.80	0.68	0.40	0.23	<1	11	-	-	4x4μ agglomerates
8	24	O_2-free	7.0	6.5	1.80	0.75	0.49	0.94	<1	1	-	+	Mat of small particles
6	24	O_2-sat.	7.0	6.9	1.78	0.55	0.35	0.49	<1	<1	-	+	Agglomerated particles
11	30	O_2-free	7.0	6.9	1.85	1.05	0.05	0.46	<1	7.5	-	+	Irregular, agglomerated particles
9	30	O_2-sat.	7.0	7.1	1.79	1.10	0.36	0.62	<1	<1	-	+	Irregular, agglomerated particles
14	36	O_2-free	7.0	7.5	1.80	1.42	~0	0.39	<1	<1	-	-	Irregular, agglomerated particles
10	36	O_2-sat.	7.0	7.4	1.80	1.12	0.54	4.31	<1	<1	-	+	Irregular, agglomerated particles
15	42	O_2-free	7.0	7.0	1.90	0.96	0.37	0.65	<1	6	-	-	Agglomerated particles
12	42	O_2-sat.	7.0	7.1	1.60	0.96	0.86	1.13	<1	8	-	+	Agglomerated particles
17	48	O_2-free	7.0	7.2	1.80	1.30	0.32	1.10	<1	<1	-	-	Mat of small particles
13	48	O_2-sat.	7.0	7.4	1.70	1.13	0.28	0.86	<1	<1	-	+	Agglomerated particles
18	84	O_2-free	7.0	6.6	2.20	1.15	0.0	0.47	<1	<1	-	-	Agglomerated particles
16	84	O_2-sat.	7.0	6.8	1.70	1.08	0.58	0.54	<1	<1	-	+	Agglomerated particles

TABLE 4

DATA INDICATIVE OF THE STORABILITY OF WATER IN INCONEL 718 CONTAINERS

Container Number	Exposure Period Months	Type of Water	Water Characterization: pH Initial	pH Final	Resistance megohm/cm Initial	Resistance megohm/cm Final	Silting Index Initial	Silting Index Final	Total Solids Content, mg/l Initial	Total Solids Content, mg/l Final	Biological Activity Initial	Biological Activity Final	Particulate Matter Characteristics
1	6	O_2-free	7.0	7.5	1.70	0.58	0	0.42	<1	<1	-	-	1 to 2μ Particles agglomerated to 30μ
2	6	O_2-sat.	7.0	7.4	1.70	0.95	0.28	0.83	<1	4	-	-	1 to 2μ Particles agglomerated to 30μ
6	12	O_2-free	7.0	7.8	1.75	0.88	0	0.14	<1	<1	-	-	Mat of small particles, 18μ agglomerates
4	12	O_2-sat.	7.0	7.3	1.80	0.98	0	0.59	<1	<1	-	X	1.3μ Particles, 50 x 90μ agglomerates
I-4	18	O_2-free	7.0	7.7	1.75	1.04	0.32	0.00	<1	5	-	-	2x2μ to 8x16μ Agglomerated irregular particles
I-1	18	O_2-sat.	7.0	7.5	1.75	0.88	0.56	0.35	<1	<1	-	-	2x2μ to 8x8μ Agglomerated irregular particles
I-2	24	O_2-free	7.0	7.3	1.80	1.00	0.15	0.11	<1	6	-	-	24x24μ Agglomerated particles
7	24	O_2-sat.	7.0	7.3	1.80	1.03	0.40	0.0	<1	3.5	-	-	48x112μ Agglomerated particles
I-5	30	O_2-free	7.0	7.2	1.80	1.09	$\sim$0	0.40	<1	5	-	-	Agglomerated 1-2μ particles
I-3	30	O_2-sat.	7.0	7.2	1.75	0.95	0.02	0.45	<1	11	-	-	Agglomerated 1-4μ particles
I-8	36	O_2-free	7.0	7.2	1.90	1.00	$\sim$0	$\sim$0	<1	5.5	-	-	Agglomerated 1-2μ particles
9	36	O_2-sat.	7.0	7.1	1.70	0.98	0.41	0.10	<1	5.5	-	-	Agglomerated 1-2μ particles
I-12	42	O_2-free	7.0	7.0	1.70	0.98	0.32	0.24	<1	4.0	-	-	Agglomerated 1-8μ particles
I-10	42	O_2-sat.	7.0	7.0	1.70	0.98	0.15	0.65	<1	2.0	-	-	Agglomerated 1-4μ particles
16	48	O_2-free	7.0	7.3	1.70	1.10	0	$\sim$0	<1	4.5	-	-	Agglomerated 1-4μ particles
I-13	48	O_2-sat.	7.0	7.4	1.70	0.95	0.33	0.04	<1	<1	-	-	Agglomerated 1-8μ particles
19	84	O_2-free	7.0	7.3	1.95	0.92	0.39	0.03	<1	<1	-	-	Many 1μ and smaller particles
I-15	84	O_2-sat.	7.0	6.6	1.70	0.96	0.54	0.05	<1	<1	-	-	Many 1μ and smaller particles

TABLE 5

DATA INDICATIVE OF THE STORABILITY OF WATER IN 17-4 PH STAINLESS STEEL CONTAINERS

Container Number	Exposure Period Months	Type of Water	Water Characterization: pH Initial	pH Final	Resistance megohm/cm Initial	Resistance megohm/cm Final	Silting Index Initial	Silting Index Final	Total Solids Content, mg/l Initial	Total Solids Content, mg/l Final	Biological Activity Initial	Biological Activity Final	Particulate Matter Characteristics
P-11	6	O_2-free	7.0	7.4	1.80	0.82	0	0.66	<1	<1	-	-	1 to 4μ particles agglomerated to 200μ
P-1	6	O_2-sat.	7.0	7.1	1.75	0.98	0	0.42	<1	<1	-	-	1 to 4μ particles agglomerated to 300μ
P-12	12	O_2-free	7.0	7.9	2.00	1.02	0.27	0.49	<1	<1	-	X	Mat of indeterminate sized particles
P-2	12	O_2-sat.	6.7	7.6	1.80	0.70	0.03	0.62	<1	<1	-	X	3 to 14μ platelets
P-13	18	O_2-free	7.0	7.4	1.95	0.86	0.40	0.05	<1	2.5	-	-	Agglomerates range from 2x8μ to 2x24μ
P-3	18	O_2-sat.	6.9	7.1	1.70	1.02	0.19	0.09	<1	5.5	-	+	Agglomerates range from 8x8μ to 8x24μ
P-14	24	O_2-free	7.0	7.0	1.80	1.07	0.40	0.0	<1	1.5	-	-	4x10μ Irregular Platelet
P-4	24	O_2-sat.	6.8	7.2	1.80	1.12	0.14	0.0	<1	4.5	-	+	3.5 x 3.5μ agglomerated particles
P-15	30	O_2-free	7.0	6.8	1.80	0.78	0.25	0.17	<1	<1	-	-	Agglomerates of 1-2μ particles
P-5	30	O_2-sat.	6.8	6.9	1.80	0.80	0.26	0.39	<1	10	-	+	Agglomerates of 1-2μ particles
P-16	36	O_2-free	7.0	7.3	1.75	0.92	0.18	0.45	<1	<1	-	-	Agglomerates of 1-4μ particles
P-6	36	O_2-sat.	6.9	7.3	1.80	1.01	0.12	0.19	<1	<1	-	+	1-4μ particles
P-17	42	O_2-free	7.0	6.9	1.75	1.00	0.49	1.21	<1	<1	-	-	1-4μ particle and agglomerates
P-7	42	O_2-sat.	6.8	7.0	1.75	0.85	0.67	0.54	<1	<1	-	+	1-4μ particle and agglomerates
P-18	48	O_2-free	7.0	7.8	1.70	1.22	0.42	0.62	<1	<1	-	-	Mat of small particles
P-8	48	O_2-sat.	7.0	7.6	1.75	1.10	0.25	0.01	<1	<1	-	+	1-8μ particles and agglomerates
P-19	84	O_2-free	7.0	7.0	1.75	0.91	0.38	0.07	<1	<1	-	-	Agglomerates of 1-2μ particles
P-9	84	O_2-sat.	7.0	6.8	1.75	0.75	0.03	0.0	<1	<1	-	+	Agglomerates of 1-2μ particles

B. Discussion of Results (cont.)

The significant items to be noted from the data are as follows:

First, the pH values of the waters change slightly during the storage periods. If the values are averaged, the following pH values are obtained for the various containers used:

> For storage in 304 stainless steel containers, the pH value increased 0.2 for the oxygen-free water and 0.3 for the oxygen-saturated water.
>
> For storage in A-286 containers, the pH value did not change for oxygen-free water but did increase 0.2 for oxygen-saturated water.
>
> For storage in 6Al-4V titanium containers, the average pH value is unchanged for oxygen-free water and increased 0.2 for oxygen-saturated water.
>
> For storage in Inconel 718 containers, the pH value increased 0.4 for oxygen-free water and 0.2 for oxygen-saturated water.
>
> For storage in 17-4PH containers, the pH value increased 0.3 for oxygen-free water and 0.3 for oxygen-saturated water.

Secondly, the resistance values of the waters have stabilized after the initial decrease in values due to an increase in concentration of ionic species, and the final resistance values still correspond to concentration levels which contain less than one part per million of dissolved metallic ions.

Third, the Silting Index values indicate the presence of a slight amount of particulate matter in the size range of less than 5 microns in the water, but the concentration levels are insignificant with regard to the quantities of particulate matter that are required to cause clogging in flow passages. In addition, the concentration level of particles in the less than 5 micron size is not increasing with increased periods of water storage.

Fourth, there is apparently no increase in the total solids content of the waters as the period of storage increases; the values observed are sufficiently low so that the quality of the water is not imparied. The level of detection in the method used corresponds to a mg/1ℓ.

B. Discussion of Results (cont.)

Fifth, the pH values, the resistance values, and the Silting Index values are not significantly different between the various storage periods.

The particulate matter collecting on the filters appeared to be that which adhered to the container walls during the cleaning, pickling, passivation, and flushing procedures prior to filling. In summation, the waters were all suitable for use in transpiration coolant devices after water storage periods of up to eighty-four months.

BIOLOGICAL CHARACTERIZATION

A. PROCEDURES

200 ml samples of the water taken from the storage containers were filtered through pre-sterilized filter pads which were transferred directly to sterilized Petri dishes containing suitable nutrients for direct colony counting after a suitable culturing period. The procedures are described in Standard Methods of Analysis of Water and Waste Water, American Public Health Association, 13th Edition (1971) and Biological Analysis of Water and Waste Water, AM 302, Millipore Corporation, Bedford, Mass. (1973). In addition, any biological organisms present were washed from the filter surfaces with a sterile buffer solution and placed directly in sterile nutrient solutions for culturing. This is to assure that adequate samples are available for identifying the genus and the specific species of microorganisms that might be present in the stored water. Based on the lag-period, prior to growth, of microorganisms which has been observed earlier in the program (Reference 1), the tubes containing the nutrient solutions were incubated for periods of up to one month.

B. DISCUSSION OF RESULTS

The results obtained by culturing samples from the water containers are presented in Tables 1 to 5 under the heading "Biological Activity." The lack of any indications of microorganisms being present is denoted by a minus sign. If growth was indicated in either the culture tubes or on the filter pad, but not on both, an "X" is used. If growth was found in both the culture tubes and on the filter pad, a plus sign is used as an indication of the positive result.

After a month of incubation of the samples from the six-month storage tests, there was no indication of microorganism growth on the pre-sterilized filter pads. Slight growth was observed in the culture tubes containing washings from the 304 stainless steel and one of the A-286

B. Discussion of Results (cont.)

containers. The number of microorganisms present was extremely small, as indicated by the negative results with the filter pads and the slight amount of growth in the culture tubes. There was no evidence that any biological growth occurred during the six-month storage period. The microorganisms found were identified as an Aeromonas species.

After a month of incubation of the samples from the twelve-month storage period, one of the culture tubes containing the washings from an Inconel 718 container exhibited growth, but the filter pad was negative. The microorganisms present were identified as most likely being Pseudomonas aeruginosa. The filter pad used for culturing the contents of both of the 17-4PH, one of the 304 stainless steel and one of the A-286 containers exhibited growth, but the corresponding culture tubes were all negative. The microorganisms were identified as a Pseudomonas species. Again there was no evidence that biological growth occurred during the twelve-month storage period in any of the containers.

After a month of incubation of the samples from the eighteen-month storage period, there was indication of microorganism growth on the filter pads and in culture tubes containing samples from one of the 304 stainless steel, one of the A-286, and one of the 17-4PH containers. The microorganisms were identified as a Pseudomonas species. The number of organisms present was extremely small, and there was no evidence that growth occurred during the eighteen-month storage period.

After a month of incubation of the samples from the twenty-four month storage period, there were indications of microorganism growth on the filter pads and in the culture tubes containing samples from one of the 304 stainless steel, both of the A-286, both of the 6Al-4V titanium, and one of the 17-4PH containers. The microorganisms were all of the same species and, based on the classifications used in the 8th edition of Bergey's "Manual of Determinative Bacteriology," were identified as most likely being Pseudomonas teslosteroni. In this edition, much of the former genus Aeromonas is now included in the genus Pseudomonas, and therefore the microorganisms observed in the two samples of the six-month storage period may be the same species as those identified in samples from the twenty-four month storage period.

The number of organisms present in the samples from the twenty-four month storage period was extremely low, less than 100 per ml of water, and there was no evidence that growth occurred during the storage period. The fact that all the organisms are of the same species indicates that they were probably introduced during the fill procedure and that the improved water removal procedure reduces the possibility of contamination during the sampling procedure.

B. Discussion of Results (cont.)

After a month of incubation of the samples from the thirty-month storage period, there were indications of microorganisms present on the filter pads and in culture tubes containing samples from both of the 304 stainless steel, one of the A-286, one of the 17-4PH and both of the 6Al-4V titanium containers. The number of microorganisms present was small, and there was no evidence that any growth occurred during the thirty-month storage period. The microorganisms present were identified as a Pseudomonas species.

After a month of incubation of the samples from the thirty-six month storage period, there were indications of microorganisms present on the filter pads and in the culture tubes containing samples from one of the 304 stainless steel, one of the A-286, one of the 6Al-4V titanium and one of the 17-4PH containers. The number of microorganisms present was extremely small, less than 2 per ml, and they were identified as a Pseudomonas species.

After a month of incubation of the samples from the forty-two month storage period, there were indications of microorganisms present on the filter pads and in culture tubes containing samples from one of the 304 stainless steel, both of the A-286, one of the 6Al-4V titanium, and one of the 17-4PH containers. The number of microorganisms present was small, and there was no evidence that any growth occurred during the forty-two month storage period. The micororganisms present were identified as a Pseudomonas species.

After a month of incubation of the samples from the forty-eight month storage period, there were indications of microorganisms present on the filter pads and in the culture tubes containing samples from one of the 304 stainless steel, both of the A-286, one of the 6Al-4V titanium, and one of the 17-4PH containers. The number of microorganisms present was small, and they were identified as a Pseudomonas species.

After a month of incubation of the samples from the eighty-four month storage period, there were indications of microorganisms present on the filter pads and in the culture tubes containing samples from one of the 304 stainless steel containers, one of the A-286, one of the 6Al-4V titanium, and one of the 17-4PH containers. The number of microorganisms present was small, and they were identified as the Pseudomonas species which had been identified in the previous storage periods. From the slight growth which was observed in the culture media, it can be inferred that the microorganisms are in a weakened condition.

In summation, the biological testing has shown that there is no evidence for any bioligical growth during the storage periods even though a slight number of microorganisms were introduced during the filling procedures.

CONTAINER EXAMINATION

A. PROCEDURES

After removal of the water from the containers by draining, they were vacuum-dried for a day to insure sectioning in a dry condition. The containers were then photographed to document their general appearance. Sectioning of the containers to expose the internal surfaces was accomplished by sawing without coolant to prevent contamination other than from sawing debris. Subsequent handling of the container halves was carefully performed to avoid touching the interior surfaces. After removal of the debris generated by sawing, the internal surfaces were then examined with the unaided eye and photographed to document their general appearance.

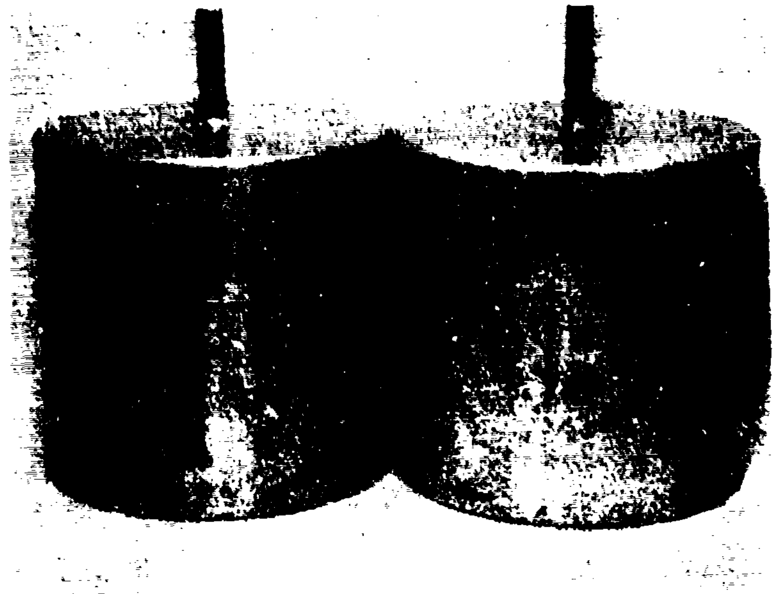
Visual examination of the interior surfaces at magnifications from 5 to 10X were conducted to define conditions found in the aforementioned visual examination and to reveal areas requiring additional examination at magnifications to 40X. All welds were examined at 40X magnification. Representative discrepancies were photographed at magnifications adequate for defect definition. Those defects requiring further definition were examined metallographically to establish their cause and extent. Sections taken either through or immediately adjacent to the affected areas were mounted, polished, and examined. Photomicrographs were taken to document the condition. All interior surfaces were dye-penetrant inspected to determine whether any defects were undetected during the visual examination.

B. DISCUSSION OF RESULTS

The results of the container examinations are discussed under three headings: (1) General Visual Examination, (2) Metallographic Examination, and (3) Implications of the Results of the Examinations.

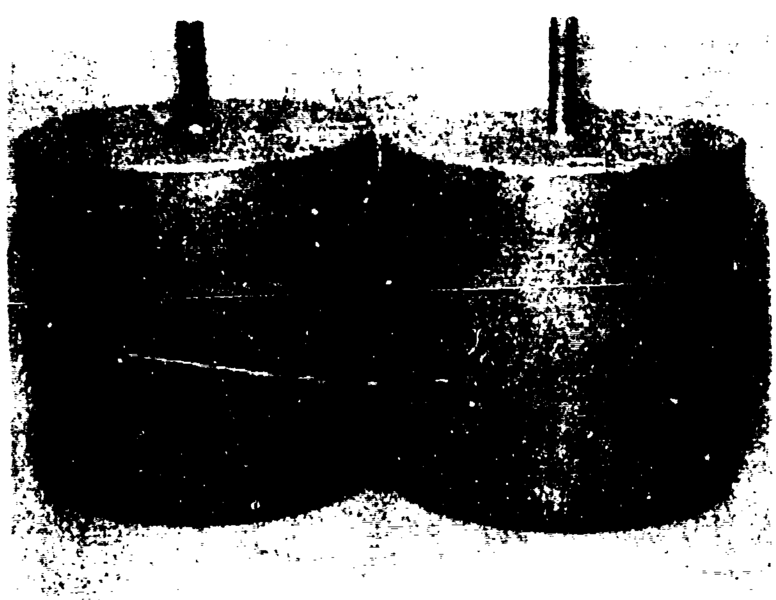
1. General Visual Examination

The external appearance of the containers is documented photographically in Figures 1 through 5. The internal appearance of the containers is documented photographically in Figures 6 through 10. Examination of these surfaces without visual aids showed full penetration for the full length of all weldments. Other conditions resulting from fabrication and cleaning procedures were as follows: (1) Pickling smut on the interiors of the 17-4PH stainless steel and Inconel 718 containers; (2) an adherent agglomeration of 17-4PH stainless steel particles in the 17-4PH stainless steel containers; (3) knifeline attach at the edge of the Inconel 718 welds as a result of pickling; (4) etching of the parent metal and welds in Inconel 718 containers and of the welds in the 17-4PH containers as a result of pickling; (5) residual oxides from heat treatment in the A-286 and titanium 6Al-4V containers; (6) rough surface of the 17-4PH stainless steel containers as a result of scale removal during pickling; and (7) stains and localized pickling attacks around areas of pre-heat-treatment contamination.



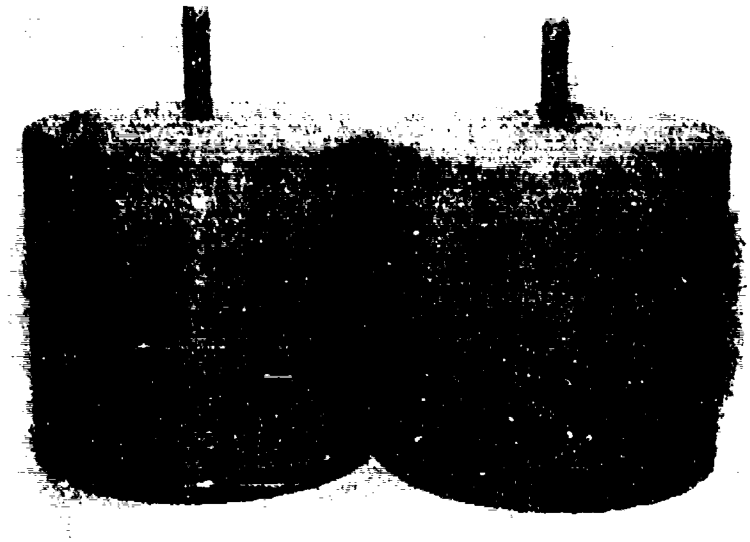
No. 16 No. 18

Figure 1. 304 Stainless Steel Containers, Magnification 1/3X



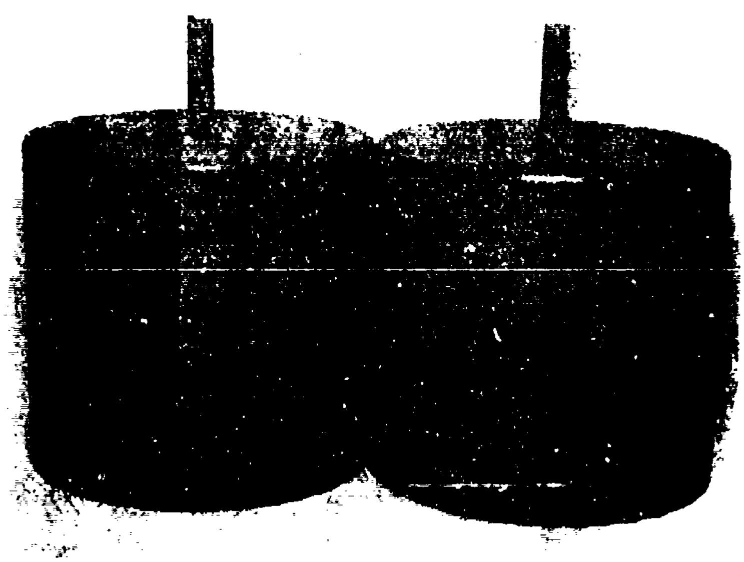
No. 16 No. 17

Figure 2. A-286 Containers, Magnification 1/3X



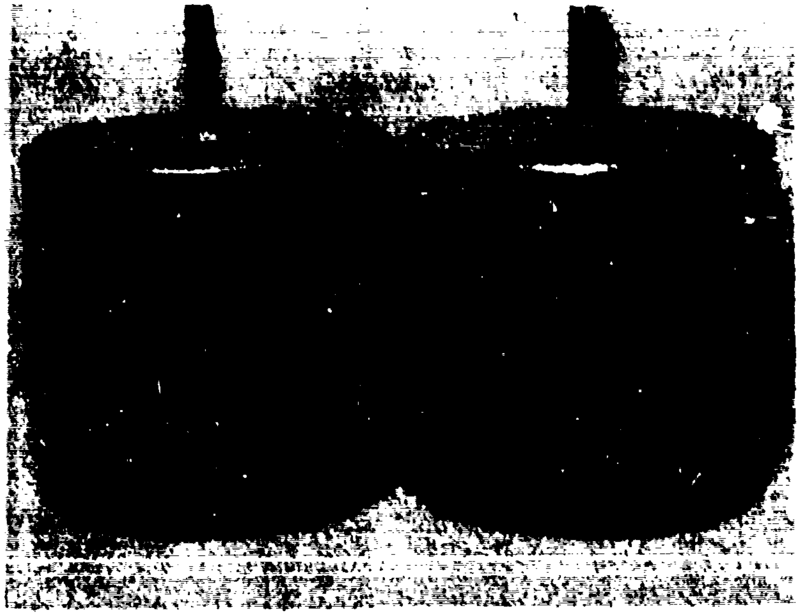
No. P9 No. P10

Figure 3. 17-4PH H-1025 Stainless Steel Containers, Magnification 1/3X



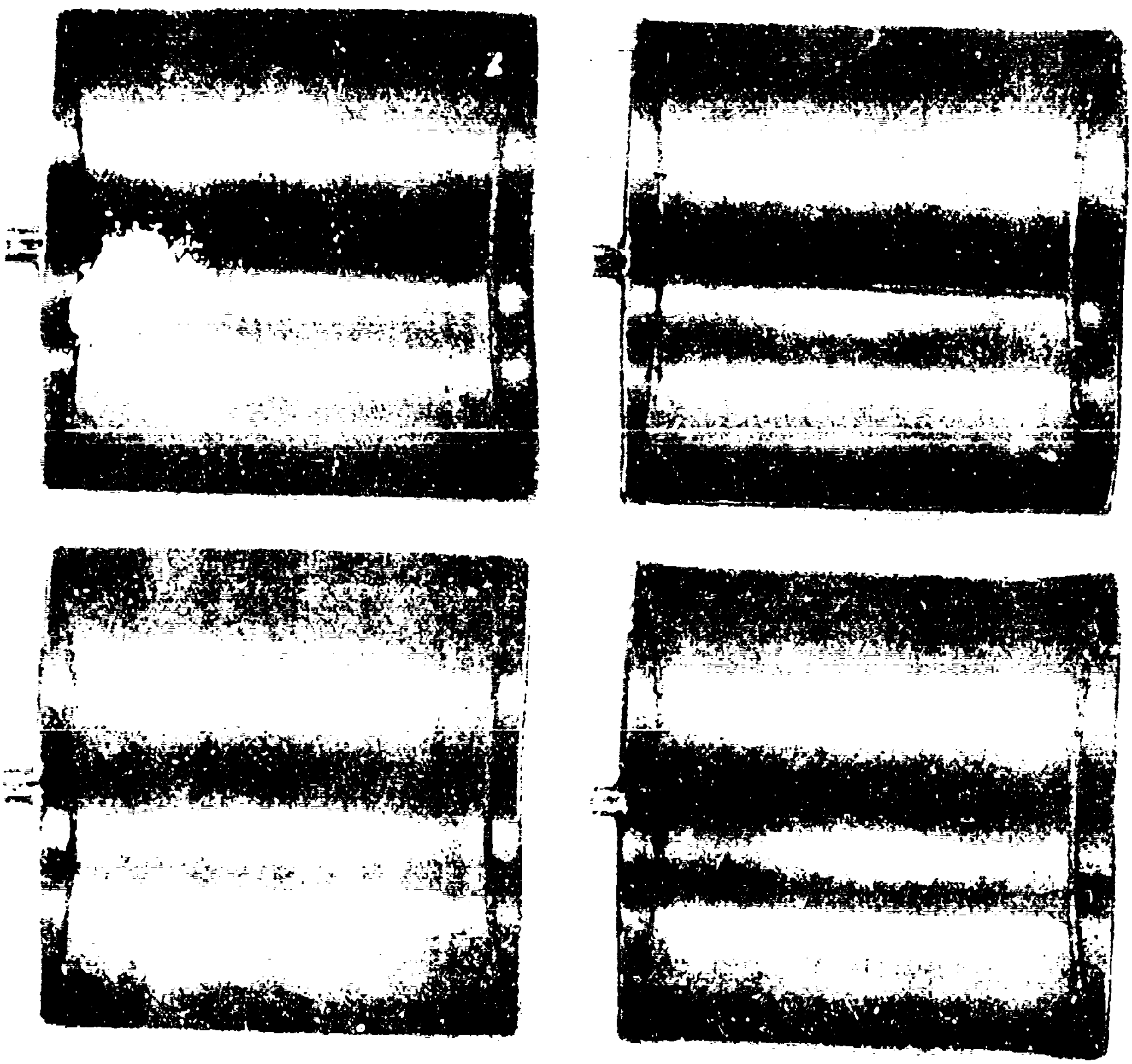
No. 16 No. 18

Figure 4. Inconel 718 Containers, Magnification 1/3X



No. 16 No. 18

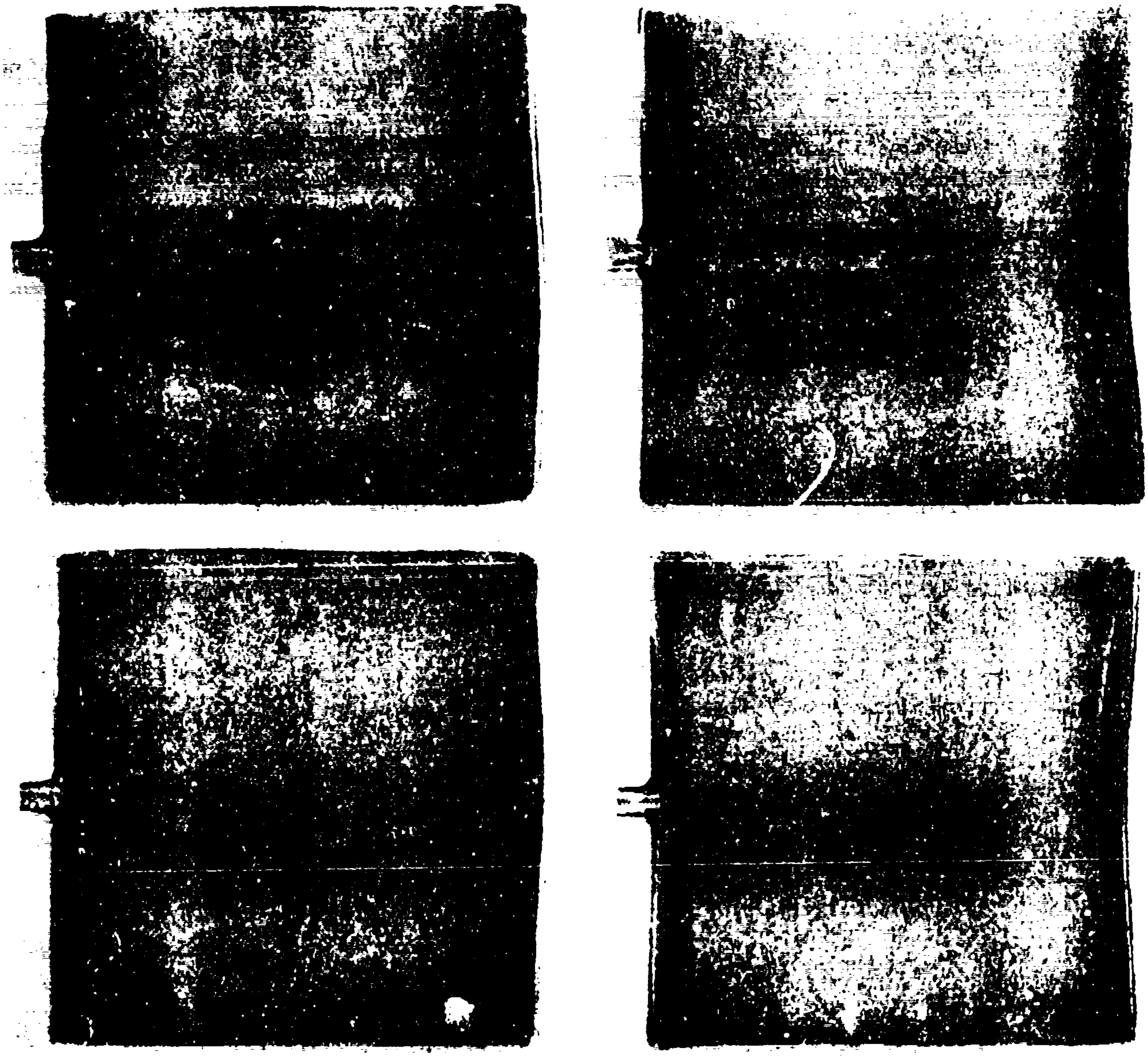
Figure 5. 6Al-4V Titanium Containers, Magnification 1/3X



No. 16

No. 18

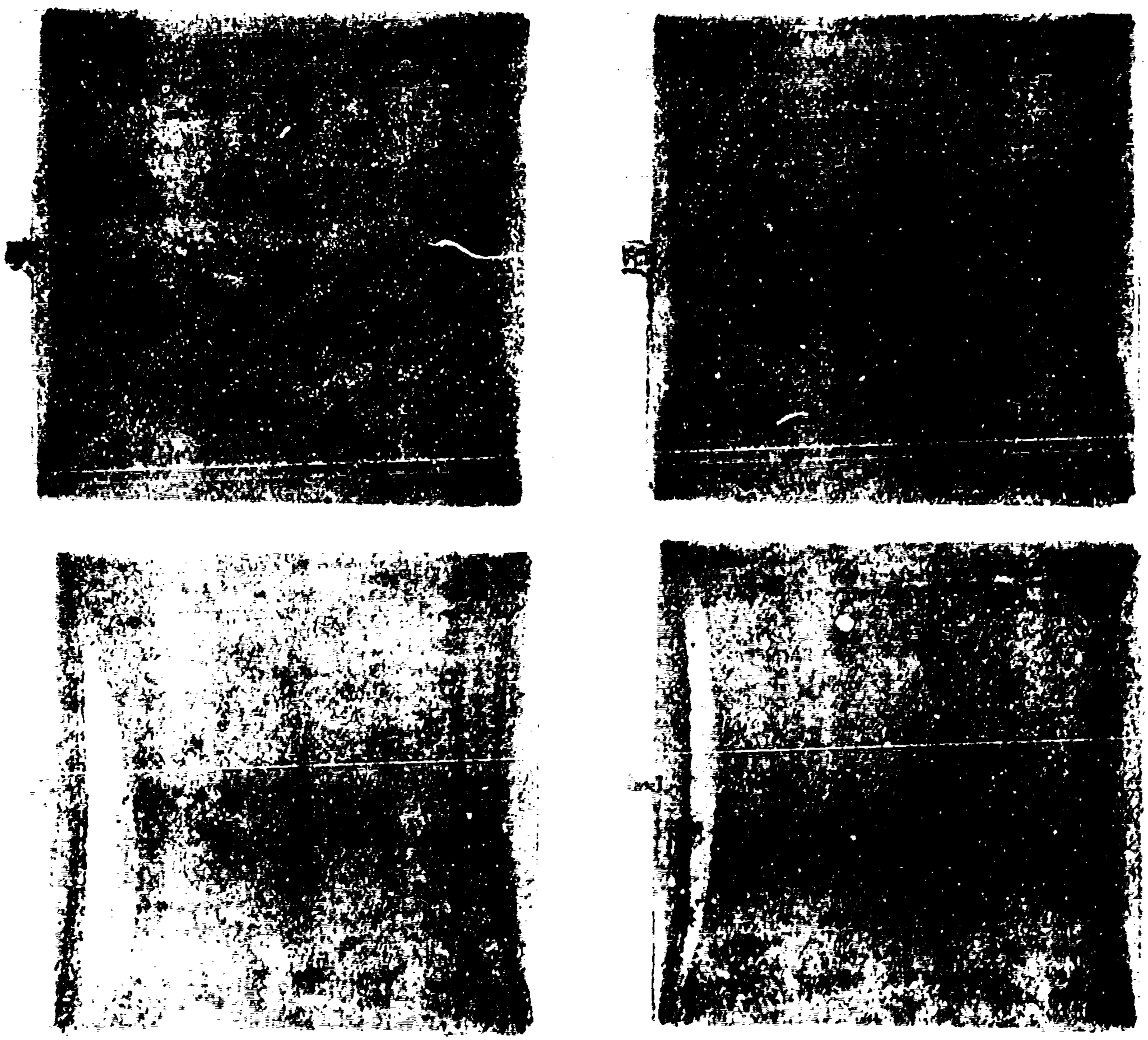
Figure 6. Interior of 304 Stainless Steel Containers, Magnification 3/4X



No. 16

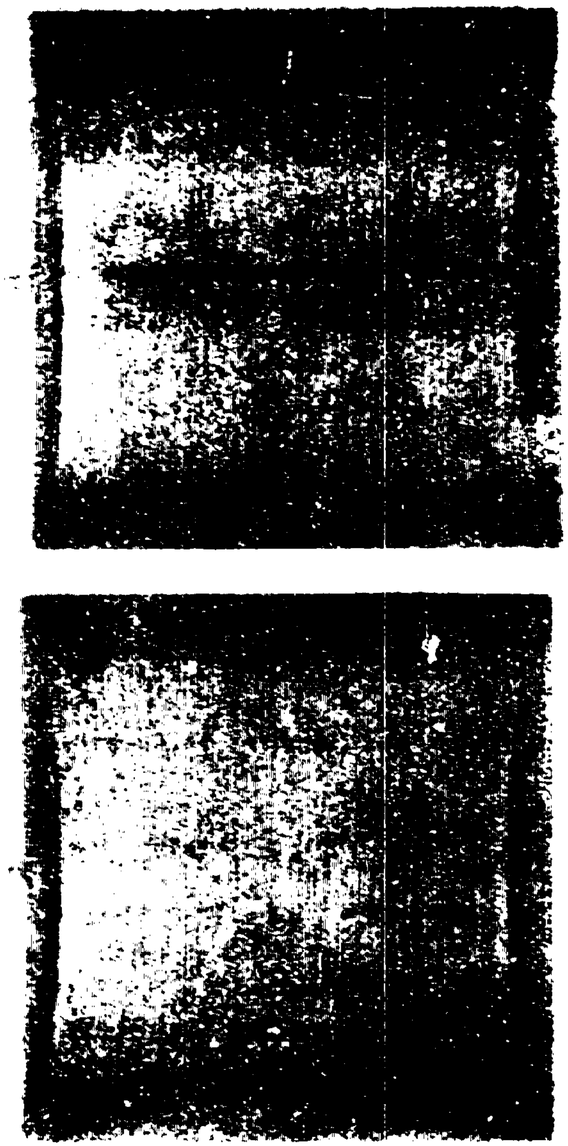
No. 17

Figure 7. Interior of A-286 Containers, Magnification 3/4X

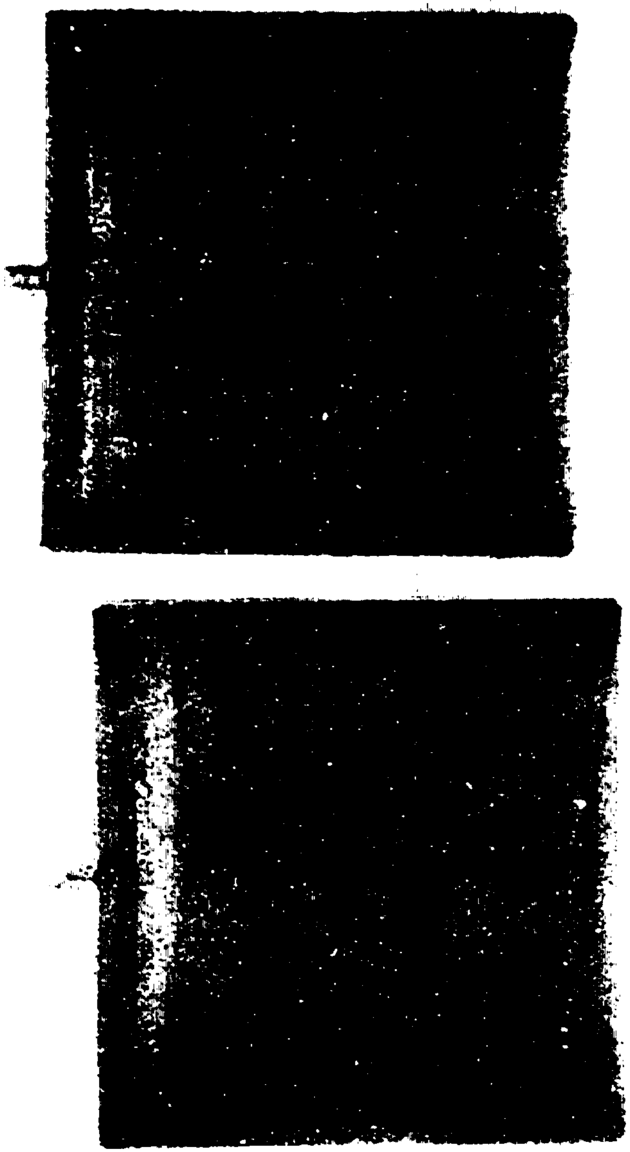


No. P9 No. P10

Figure 8. Interior of 17-4PH H-1025 Stainless Steel Containers, Magnification 3/4X

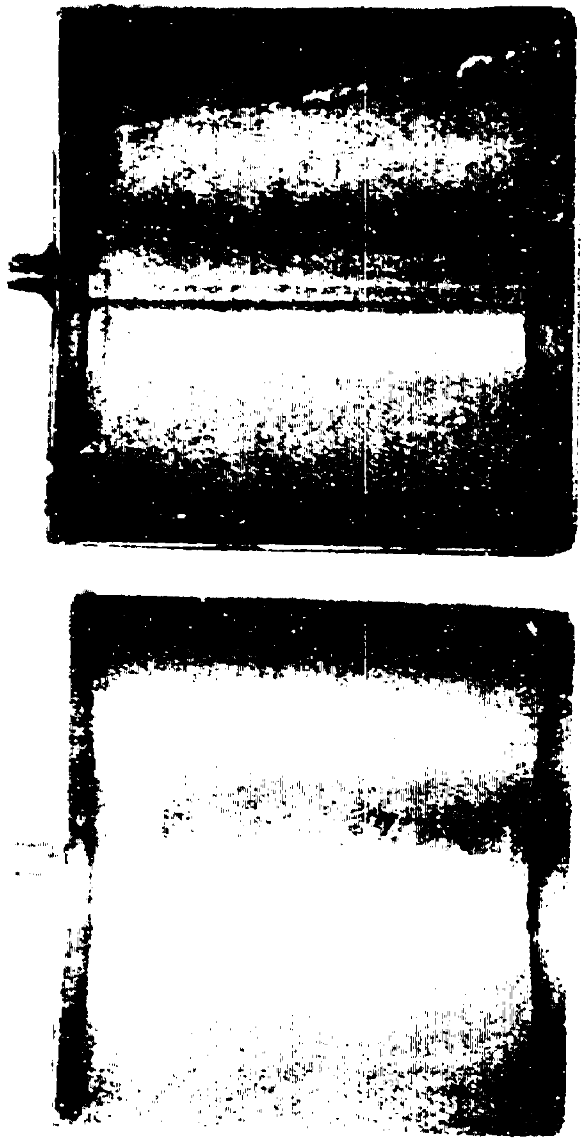


No. I-15



No. 17

Figure 9. Interior of Inconel 718 Containers, Magnification 3/4X



No. 16

No. 18

Figure 10. Interior of 6Al-4V Titanium Alloy Containers, Magnification 3/4X

B. Discussion of Results (cont.)

No significant differences were apparent between the six, twelve, twenty-four, thirty, thirty-six, forty-two, forty-eight, and eighty-four month exposure containers, or between those holding the oxygen-free and oxygen-saturated water.

2. Metallographic Examination

A summary of the analyses performed on the containers is presented in Table 6. The results of the macro- and micro-examinations are shown in Figures 11 and 17. The results of the analyses are discussed below for each container material.

a. 304 Stainless Steel

The interiors of the containers were in excellent condition, exhibiting no significant defects.

b. A-286

One weld shrinkage crack was found in the two containers. This crack, which is similar to those found in previously examined containers, is shown in Figure 11. A photomicrograph of the crack is shown in Figure 15. Light residual oxide remains at the top of the containers after pickling. The oxide appears as the dark area on the left side of the container in Figure 7. No evidence of corrosion resulting from the storage of water could be found.

c. 17-4PH Stainless Steel

These containers exhibited the same conditions found in the previously examined containers, i.e., smut, roughening of the interior surfaces due to scale removal during pickling, and tightly adherent agglomerated 17-4PH stainless steel particles which are deposited in isolated areas of corrosive attack by pickling. These particles have been previously identified as 17-4PH stainless steel; like smut, they are considered to be reaction products of the pickling operation. This condition is shown in Figure 12. No evidence of corrosion from the storage of water could be found.

d. Inconel 718

As with the previously examined containers, examination of the interior surfaces at moderate magnification revealed knifeline corrosive attack at the edge of all closure weldments. This condition, which is attributed to pickling, is shown in Figure 13 as the dark line at the bottom edge of the weld. A photomicrograph of a section taken through

TABLE 6

SUMMARY OF CONTAINER ANALYSIS

Material	Container Iden.	5-10X Inspection	40X Inspection	Dye-Penetrant Inspection*	Metallographic Documentation Macro	Metallographic Documentation Micro
304 Stainless Steel	16	Bright Finish	-	No Indications		
	16W	Bright Finish	-	No Indications		
		Bright Finish	-	No Indications		
		Bright Finish	-	No Indications		
A-286	16	Dull Finish, Oxide at Top	One Weld Crack	No Additional Indications	X	X
	16W	Dull Finish	-	No Indications		
	17	Dull Finish	-	No Indications		
	17W	Dull Finish	-	No Indications		
17-4PH Stainless Steel	P-19	Smut, Rough Surface from Scale Removal, Welds Etched, Agglomerated 17-4PH Particles	-	No Indications		
	P-19W	Smut, Rough Surface from Scale Removal, Welds Etched, Agglomerated 17-4PH Particles	-	No Indications		
	P-9	Smut, Rough Surface from Scale Removal, Welds Etched, Agglomerated 17-4PH Particles	-	No Indications	X	
	P-9W	Smut, Rough Surface from Scale Removal, Welds Etched, Agglomerated 17-4PH Particles	-	No Indications		

TABLE 6 (cont.)

Material	Container Ident.	5-10X Inspection	40X Inspection	Dye-Penetrant Inspection*	Metallographic Documentation Macro	Metallographic Documentation Micro
Inconel 718	I-15	Smut, Parent Metal and Weld Etched	Knifeline Pickling Attack at Edge of Weld	No Indications		
	I-15W	Smut, Parent Metal and Weld Etched	Knifeline Pickling Attack at Edge of Weld	No Indications		
	19	Smut, Parent Metal and Weld Etched	Knifeline Pickling Attack at Edge of Weld	No Indications		
	19W	Smut, Parent Metal and Weld Etched	Knifeline Pickling Attack at Edge of Weld. One Weld Crack	No Additional Indications	X	X
Titanium 6Al-4V	16	Isolated-Light Blue Oxide at Top, Stained Areas and Isolated Pickling Attack Around Area of Pre-Heat Treatment Contamination	-	No Indications		
	16W	Isolated Light-Blue Oxides at Top, Stained Areas and Isolated Pickling Attack Around Areas of Pre-Heat Treatment Contamination	-	No Indications	X	
	18	Isolated Light-Blue Oxides at Top, Stained Areas and Isolated Pickling Attack Around Areas of Pre-Heat Treatment Contamination	-	No Indications		
	18W	Isolated Light-Blue Oxides at Top, Stained Areas and Isolated Pickling Attack Around Areas of Pre-Heat Treatment Contamination	-	No Indications		

"W" Indicates the container half with weld in cylindrical portion.
* Dye-penetrant indications exclude those found visually.

Figure 11. Weld Crack in A-286 Container, Magnification 14X

Figure 12. Adherent, Agglomerated 17-4PH Stainless Steel Particles Found in the 17-4PH Stainless Steel Containers, Magnification 14X

Figure 13. Weld Crack in Inconel 718 Container, Magnification 14X

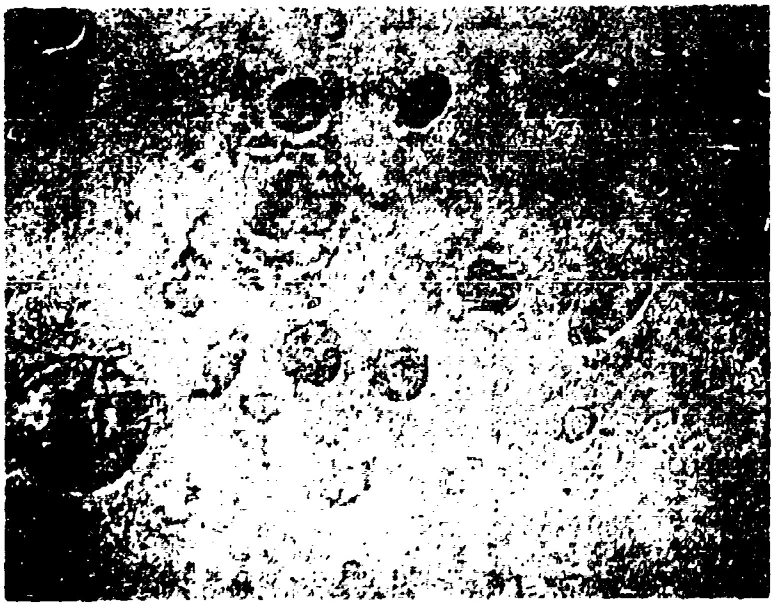


Figure 14. Isolated Pickling Attack Around Areas of Contamination in Titanium 6Al-4V Container, Magnification 14X

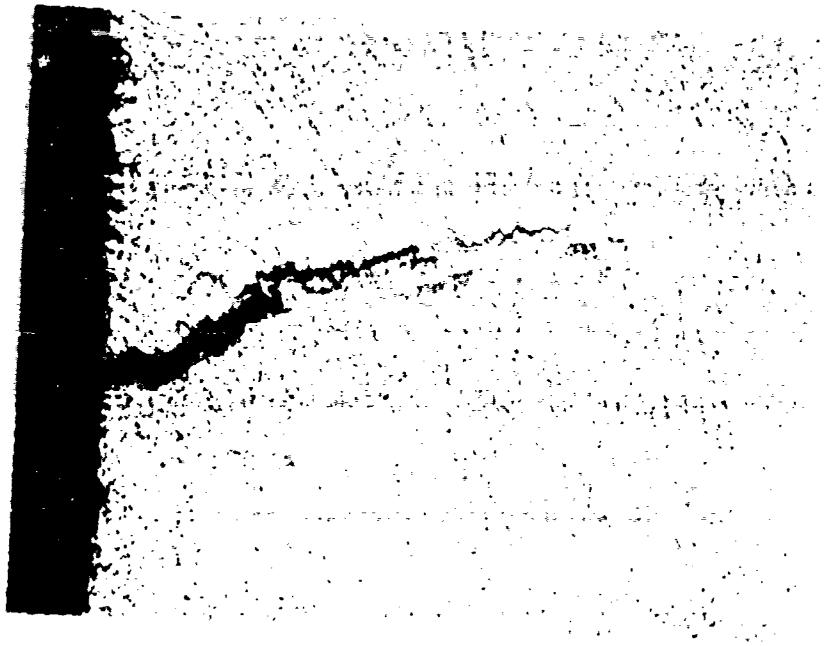


Figure 15. Photomicrograph of Weld Crack in A-286 Container, Magnification 100X

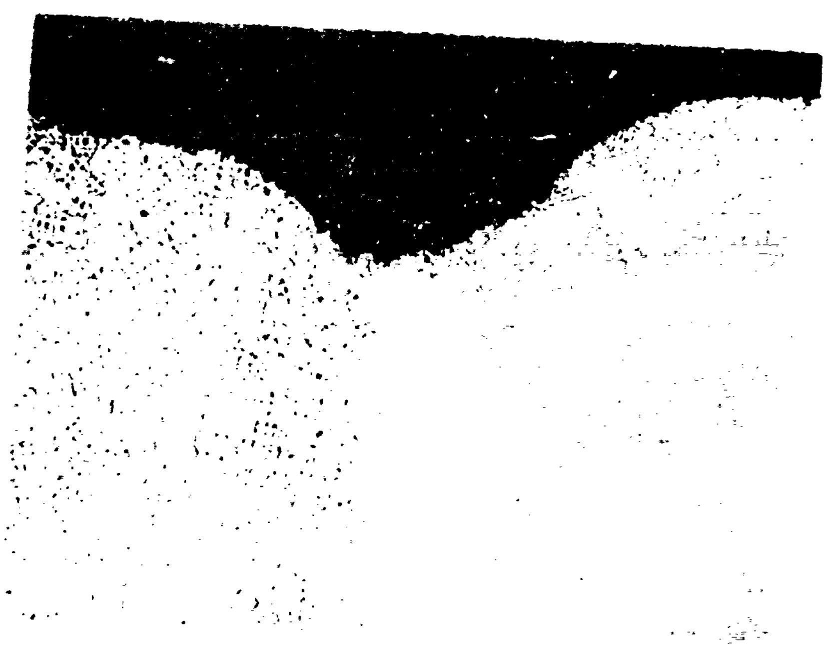


Figure 16. Photomicrograph of Localized Pickling Attack at Edge of Inconel 718 Weld, Magnification 100X

Figure 17. Photomicrograph of Weld Crack and Shrink Porosity in Inconel 718 Weld. The Weld Surface (Top) Shows Interdentritic Attack from Pickling. Magnification 100X

this area is shown in Figure 16. The one weld crack found during the examination is shown in Figure 13. A photomicrograph of the crack is shown in Figure 17. The photomicrograph also reveals interdendritic attack from pickling at the weld surface and shrink porosity in the weld interior. The containers displayed a tightly adherent smut as a result of pickling. No anomalies attributable to water storage were found.

e. **Titanium 6Al-4V**

The container interior surfaces exhibited small isolated areas of light-blue oxide and stains and corrosive attack from pickling around areas of contamination present during heat treatment. The residual oxides formed during heat treatment (aging) in air and were not removed during pickling. It appears that the presence of contamination during heat treatment contributed to oxide resistance to removal by pickling. As shown in Figure 14, some contaminated areas produced corrosion attack on their periphy during pickling. All defects found in the containers are due to inadequate cleaning prior to heat treatment. No effects attributable to the storage of water were found.

3. <u>Implications of the Results of the Examination</u>

Discrepancies found in the internal surfaces of the containers were due to fabrication and cleaning procedures rather than the quality of the stored water. The discrepancies were the same as those found in previously examined storage containers. The most serious of these discrepancies, with regard to influencing the function of the containers for storing transpiration coolant water, is the generation of a smut on the Inconel 718 and 17-4PH stainless steel during pickling. However, none of these irregularities have produced particles of sufficient size and quantity to degrade the functional quality of the water. The primary cause of these conditions is attributed to poor inert gas coverage on the container interiors during heat treatment and to the resulting excessive oxidation.

The presence of contaminants in the titanium alloy containers prior to heat treatment and in the weld cracks found in the A-286 and Inconel 718 containers is attributable to inadequate process control on the part of the container supplier. In spite of the presence of the contaminants, the functional quality of this water was not impaired.

SECTION III

CONCLUSIONS AND RECOMMENDATIONS

A. CONCLUSIONS

The following conclusions are drawn from the eighty-four month storage evaluation program.

1. All five container materials, 304 and 17-4PH stainless steels, A-286, Inconel 718, and 6Al-4V titanium, are suitable for the storage of water for transpiration cooling purposes.

2. Both the oxygen-free and oxygen-saturated waters demonstrated favorable storability characteristics with regard to pH changes, ionic contamination as evidenced by electrical resistance measurements, and particulate formation as evidenced by Silting Index values and microscopic inspection of filters.

3. Based on the biological tests, there is no evidence that biological growth has occurred in the containers during storage periods up to eighty-four months.

4. Discrepancies in the containers, such as cracks, smut and etched surfaces, were produced during fabrication, cleaning, and passivation of the containers. Yet with adequate flushing prior to storage, the stored water is suitable for use as a transpiration coolant.

B. RECOMMENDATIONS

1. Long-term storage tests should be conducted with the selected waters in containers in which dissimilar metals are present to demonstrate which combinations of metallic materials can be used as suitable components in a transpiration coolant system.

2. The ease of fabrication, cleaning, and passivation of containers should be given proper priority in the selection and design of container materials for transpiration coolant devices.

3. Long-term storage tests should be conducted with the selected waters in containers which contain non-metallic materials which may be of use in the fabrication of transpiration coolant systems.

APPENDIX A

FABRICATION AND TREATMENT PROCEDURES FOR WATER CONTAINERS

FABRICATION AND TREATMENT PROCEDURES FOR WATER CONTAINERS

The fabrication procedures, the heat treatment cycles, the cleaning procedures, and the passivation procedures for the water containers prior to filling are presented below.

A. CONTAINER FABRICATION

Drawings of the long-term storage container and its associated weld tooling are shown in Figures A-1, A-2, and A-3. The sequence of operations in the container fabrication is as follows:

1. Cylindrical Tube, -1

a. Fabricate -1 cylinder tube by rolling the sheared sheet stock into the desired diameter.

b. Weld the lingitudinal joint using the automatic GTA* welding process. Full weld penetration must be obtained using gas backup for the welds.

c. Dye-penetrant inspect welds, then trim tubes to required length. No cracks allowed.

d. Machine tube ends for weld joint preparation.

e. Clean tubes and store in appropriate container while awaiting next assembly.

2. Fill-Tube, -2

a. Section fill-tubes to 6 ± 0.12 in. lengths.

b. Deburr fill-tube ends and inspect.

c. Clean fill-tubes and then package individually and store for next assembly.

3. Tank Head, -3

a. Blank tank heads to the desired diameter by punching or sawing and then machining.

b. Machine weld joint preparation at outer edge as required.

c. Drill the fill-tube hole to mate with the fill tube.

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*Gas Tungsten Arc

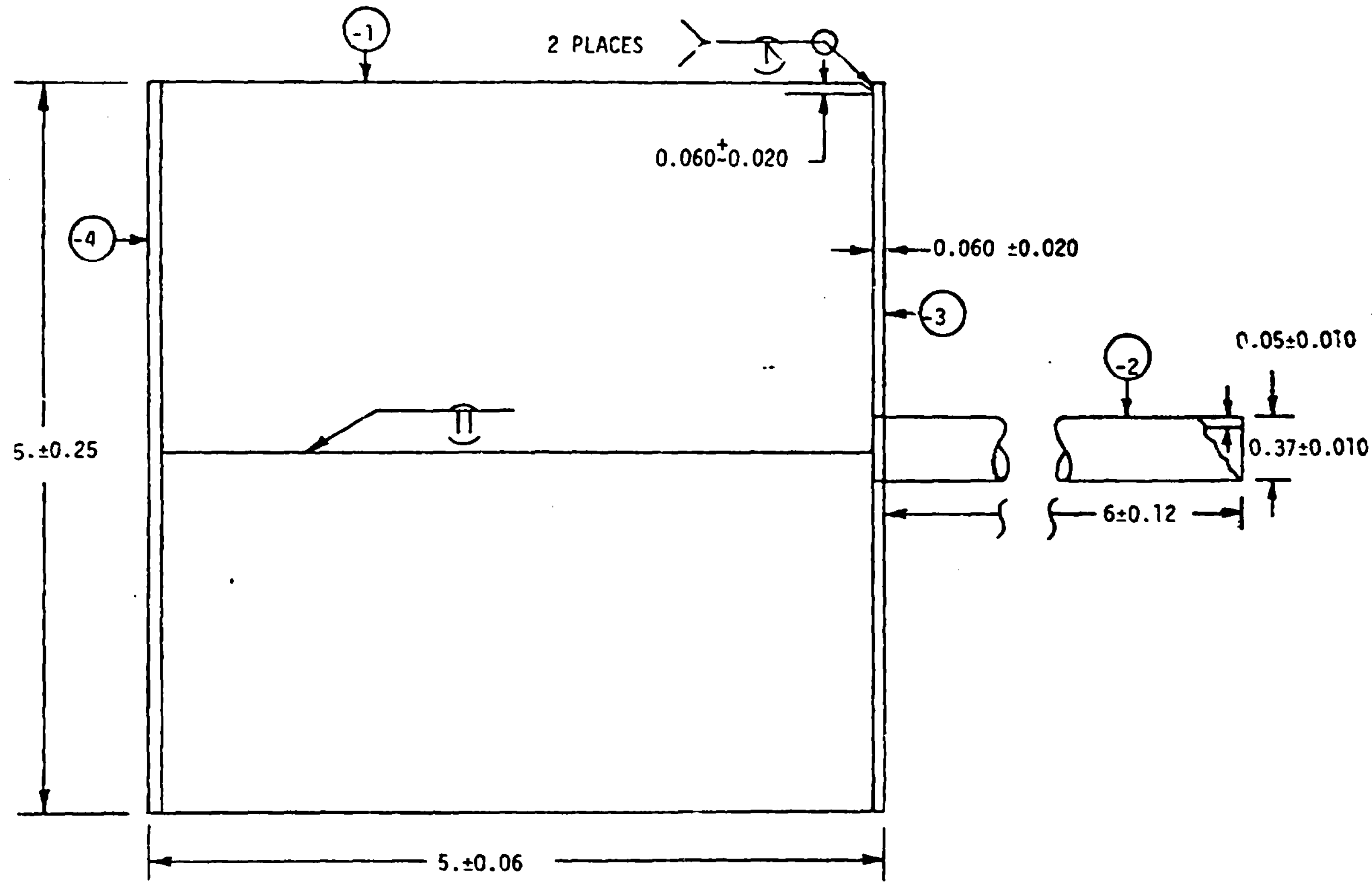

Figure A-1. Long-Term Storage Container

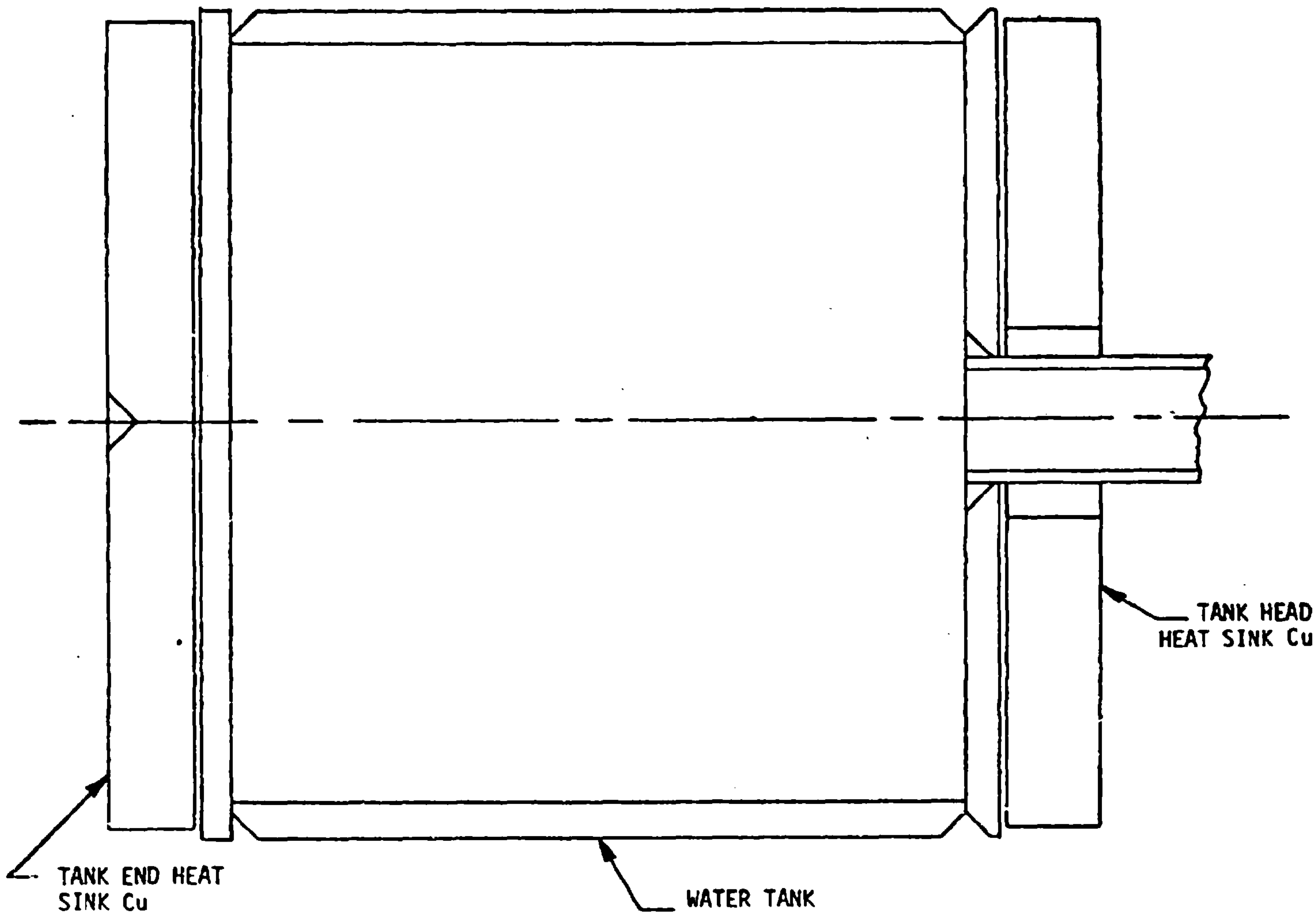

Figure A-2. Weld Tooling for Tank Closures

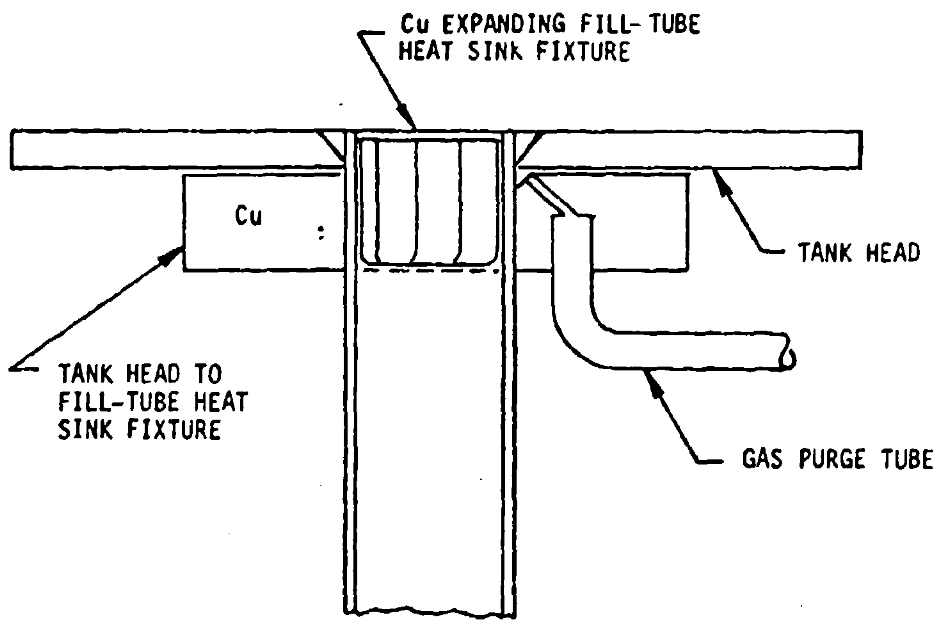

Figure A-3. Weld Tooling for Fill-Tube to Tank Head Joint

A, Container Fabrication (cont.)

d. Counter bore the tank head on one side at the fill-tube hole for weld joint preparation.

e. Inspect tank heads, clean and package.

4. Tank End, -4

a. Perform operations 3,a, 3,b, and 3,e as above.

5. Assembly Sequence

a. Assemble -2 fill-tube into -3 tank head using appropriate weld fixture and tooling.

b. Weld root pass on grooves side without weld filler wire by the automatic GTA welding process.

c. Clean root pass by rotary wire brushing using a clean stainless steel wire brush.

d. Weld the cover pass using the appropriate weld filler wire.

e. Reposition the part in the weld fixture and then place a filler weld at the fill-tube to tank head junction.

f. Inspect welds and clean part.

g. Fixture tank head assembly with -1 tube for welding and purge tank with Ar or He.

h. Weld root pass without the use of weld filler wire. Full penetration must be obtained at the ID of the joint.

i. Dye-penetrant inspect and clean root pass. No cracks allowed.

j. Fill weld groove using the appropriate weld filler wire.

k. Inspect and clean the inner tank surfaces.

l. Locate the tank end to the tank for welding, then purge the closed tank with Ar or He.

A, Container Fabrication (cont.)

m. Make the root pass without the use of weld filler wire to insure full penetration.

n. Clean root pass surface.

o. Inspect the internal weld penetration using a borescope. Full weld penetration is required.

p. Run weld cover pass using the appropriate weld filler wire.

q. Clean, inspect, and package unit for shipment.

Heat treatment of the containers was performed in an argon atmosphere, and with one exception, as noted below, utilizing the thermal cycles listed in Table A-I.

Upon receipt of the tanks, the certifications were examined, and it was found that some of the material used to fabricate the 304L stainless steel containers was actually 304 stainless steel. With the concurrence of the Air Force, the containers were subjected to a heat-treatment at 1925°F in hydrogen, followed by rapid cooling to preclude sensitization in the weld heat-affected zone.

B. PREPARATIVE PROCEDURES

1. Container Cleaning and Passivation

All the containers were degreased by submerging and agitating the tanks three times in fresh isopropyl alcohol. The containers were then purged with dry, filtered nitrogen and placed in a vacuum chamber for final drying. The A-286, 17-4PH stainless steel, and Inconel 718 containers were then subjected to an alkaline descaling treatment for 60 minutes with Kelite No. 235 at a concentration level of 32 oz per gal at 190°F. The containers were then rinsed with water at 150°F for 2 to 5 minutes, followed by a rinse at ambient temperatures with deionized water for 2 to 5 minutes, then purged with dry, filtered nitrogen, and finally dried in a vacuum chamber.

The 304, A-286, and 17-4PH stainless steels and Inconel 718 were descaled in a pickling solution of 20% HNO_3, 5% HF, and 75% H_2O at 130°F for 30 minutes per immersion until the last traces of scale were removed, as confirmed by examination with a borescope. Following the acid treatment, the tanks were flushed with tap water for 2 to 5 minutes, then flushed with deionized water for 2 to 5 minutes, then subjected to ultrasonic vibration in a water bath for 15 minutes, flushed with deionized water for 5 minutes, purged with dry, filtered nitrogen, and finally dried in a vacuum chamber.

TABLE A-I

HEAT TREATMENT PROCEDURES FOR WATER CONTAINERS

Material	Weld Wire Material	Heat Treatment	Surface Finish
304L Stainless Steel	-	-	Alkaline Clean, Pickle and Passivate
304L Stainless Steel	308L	-	Alkaline Clean, Pickle and Passivate
A-286	-	Solution Treat and Age - MIL-H-6875	Alkaline Clean, Pickle and Passivate
A-286	A-286	Solution Treat and Age - MIL-H-6875	Alkaline Clean, Pickle and Passivate
17-4 PH Stainless Steel	-	Age to H-1025 Condition - MIL-H-6875	Alkaline Clean, Pickle and Passivate
17-4 PH Stainless Steel	17-4	Solution Treat and Age to H-1025 Condition - MIL-H-6875	Alkaline Clean, Pickle and Passivate
Inconel 718	-	Age (1400 - 1200°F)	Alkaline Clean, Pickle and Passivate
Inconel 718	Inconel 718	Solution Treat (1950°F) and Age (1400-1200°F)	Alkaline Clean, Pickle and Passivate
6Al-4V-Titanium	6Al-4V Titanium	Age (1000°F - 4 Hours) - Weld-Stress Relieve (1000°F - 4 Hours)	Pickle

B, Preparative Procedures (cont.)

The 304 and A-286 stainless steels and Inconel 718 were passivated with a 30% HNO_3/3% $Na_2Cr_2O_7$ aqueous solution at 130°F for 25 minutes, rinsed with tap water for 2 to 5 minutes, rinsed with deionized water for 2 to 5 minutes, subjected to ultrasonic vibration in water for 5 minutes, rinsed with deionized water for 2 to 5 minutes, purged with dry, filtered nitrogen, and dried in a vacuum chamber. The 17-4PH stainless steel received the same treatment with the exception of a 10-minute passivation time.

The 6Al-4V titanium was degreased in isopropyl alcohol as described previously for the other tankage and then descaled in a pickling solution of 33.2% HNO_3, 1.6% HF, and 65.2% water at 140°F for 3 minutes. The titanium containers were rinsed with 130°F tap water for 2 to 5 minutes, followed by a rinse in deionized water for 2 to 5 minutes, then subjected to ultrasonic vibration in water for 5 minutes, rinsed with deionized water for 2 to 5 minutes, purged with dry nitrogen, and then dried in a vacuum chamber.

2. Container Sterilization, Filling, and Sealing

Following the final rinsing with deionized water and the drying of the container in a vacuum chamber, the containers were wrapped with reusable sterilization paper. The wrapped containers were then sterilized in an autoclave at 250°F with 15 psig steam for 30 minutes, followed by a 30-minute drying period. The containers were then stored in the paper to maintain their sterile condition.

All the steps required to fill the containers with water were conducted in the sterile laminar-flow bench. The tanks were removed from the wrapping paper in the laminar-flow bench. The tanks were weighed empty; then weighed when filled completely with sterile water to determine the total volume of the tank. The water was drained out and the tank was rinsed once more with the sterile water. A sample of the rinse water was checked for pH, conductivity, and Silting Index. If the values indicated that particulate matter and dissolved species were not present, the tank was considered ready for filling; if the values indicated that contaminants were present, the tank was rinsed until there was no evidence of contamination.

Before the final filling with oxygen-saturated deionized water, the tank was purged with oxygen from a filtered supply. The tank was then filled with the water and a sample was withdrawn for pH, conductivity, and Silting Index measurements. The ullage was adjusted to the ten percent value by weighing the container and its contents; the ullage space was purged with the filtered oxygen; and the container was capped with a sterile, tapered plug made from the same material as the container. The plug was seated in the fill-tube by use of a hammer. The containers were filled with the oxygen-free, deionized water in an analogous manner, except that filtered nitrogen was used instead of oxygen for purging and blanketing the container.

B, Preparative Procedures (cont.)

The final sealing of the containers was accomplished by GTA-welding the fill-tube/plug interface. The welds were inspected visually for any apparent anomalies. None were found. Then the containers were placed in plastic bags and labeled for the long-term storage tests.

The sampling plan for the long-term storage tests is to remove one container of each material with the two types of water for inspection and evaluation every six months for a period of five years. The contents will be characterized with respect to pH, conductivity, particulate content, and biological activity, and the containers themselves will be subjected to metallurgical examination if the other test data indicate that this is required.

APPENDIX B

SILTING INDEX MEASUREMENT

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SILTING INDEX MEASUREMENT

In order to obtain a measurement of the clogging tendencies of the particulate matter which may be produced during the storage of water in the presence of metals and non-metals for prolonged periods of time, a flow test of the water samples through nominal 1 micron size pores was required. Due to the limited quantity of the water available for each sample, approximately 20 ml, the ASTM Method F 52-69, "Silting Index of Fluids for Processing Electron and Micro-electronic Devices," was selected as being appropriate for the program.

The method determines the silting or clogging tendency of a fluid containing fine particles and gelatinous materials suspended in the fluid. The fluid is filtered through a membrane filter having a uniform pore size of 0.8 micron at a constant differential pressure. Particles larger than 5 microns form an open network above the filter and do not affect the clogging tendency; particles smaller than 5 microns tend to block the flow passages of the filter and cause a decay in the flowrate. The rate of flow decay is expressed in terms of a Silting Index value: the greater the value, the greater the clogging tendency. A schematic diagram of the apparatus in shown in Figure B-1.

The total volume of fluid passed through the filter is 12 ml. The flow of the last 10 ml is timed incrementally as V_1 (1 ml), V_2 (5 ml), and V_3 (10 ml), with T_1, T_2, and T_3 the times required to flow the respective volumes. The Silting Index value is calculated from the equation:

$$S.I. = \frac{T_3 - 2T_2}{T_1}$$

The tests in the program were conducted with three silting heads, No. 1 with an effective area of 1.0 mm^2, No. 2 with an effective filter area of 4.3 mm^2, and No. 3 with an effective filter area of 18.5 mm^2. The procedure used in the tests was that prescribed in ASTM Method F52-69, except that samples were tested in triplicate only when sufficient sample was available. A photograph of the apparatus* used is shown in Figure B-2.

Water which has passed through the 0.22 micron pore size absolute filter, and which was used in preparation of the tests, always produced a Silting Index value of less than 1 using the No. 1 silting head which has a cross-sectional area of 1 mm^2. The effect of particle size on the Silting Index was evaluated using latex spheres with a mean particle size of 1.25 microns with a range from 0.5 to 2.0 microns and puff ball spores with a size range from 3 to 4 microns. These data are presented for silting heads Nos. 1, 2, and 3 which have cross-sectional areas of 1 mm^2, 4.3 mm^2, and 18.5 mm^2, respectively, in Figures B-3 and B-4. The particle concentration is given in mg/l because the suspensions were prepared on a weight basis; the actual

*Available from Millipore Corp., Bedfore, MA

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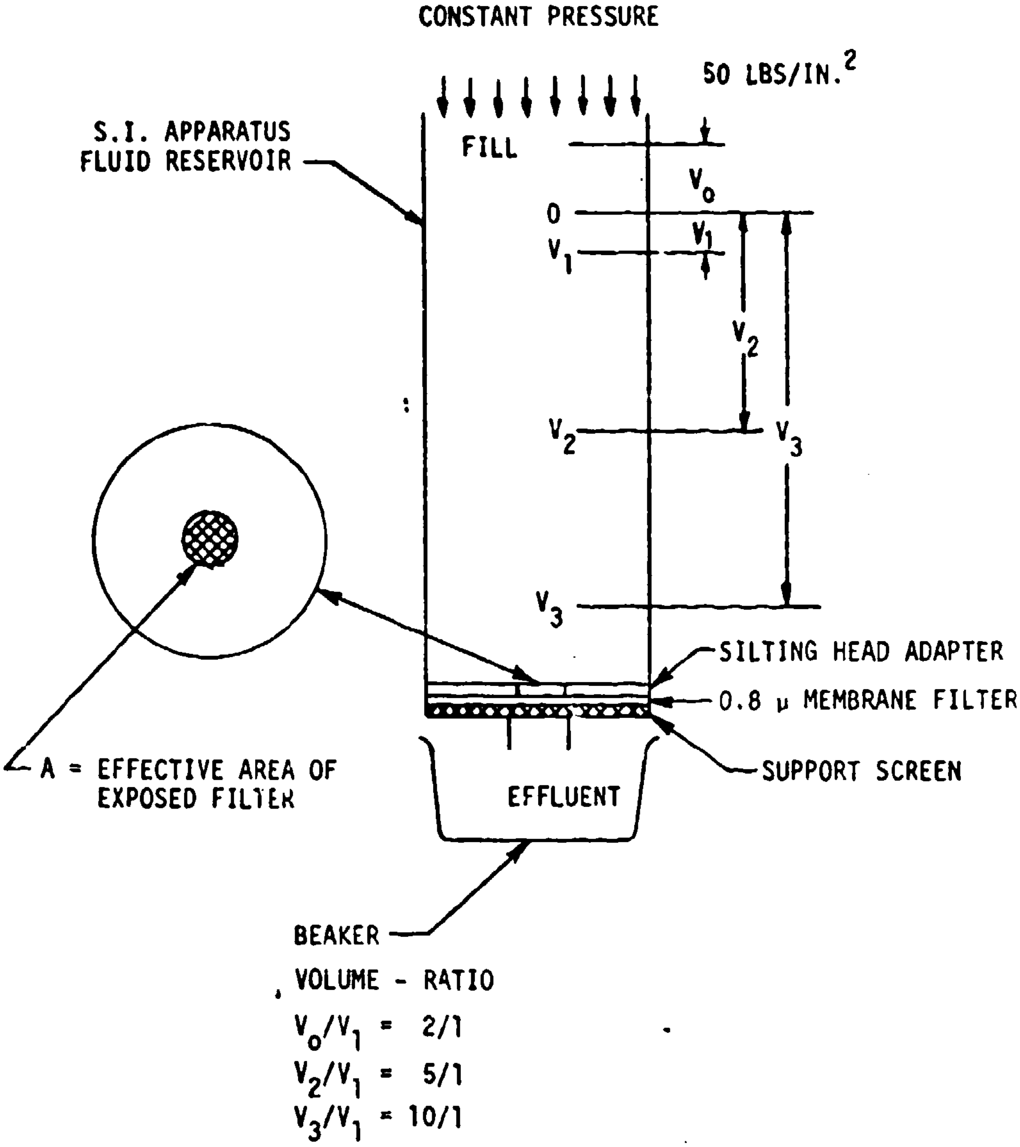

Figure B-1. Schematic of Silting Index Apparatus

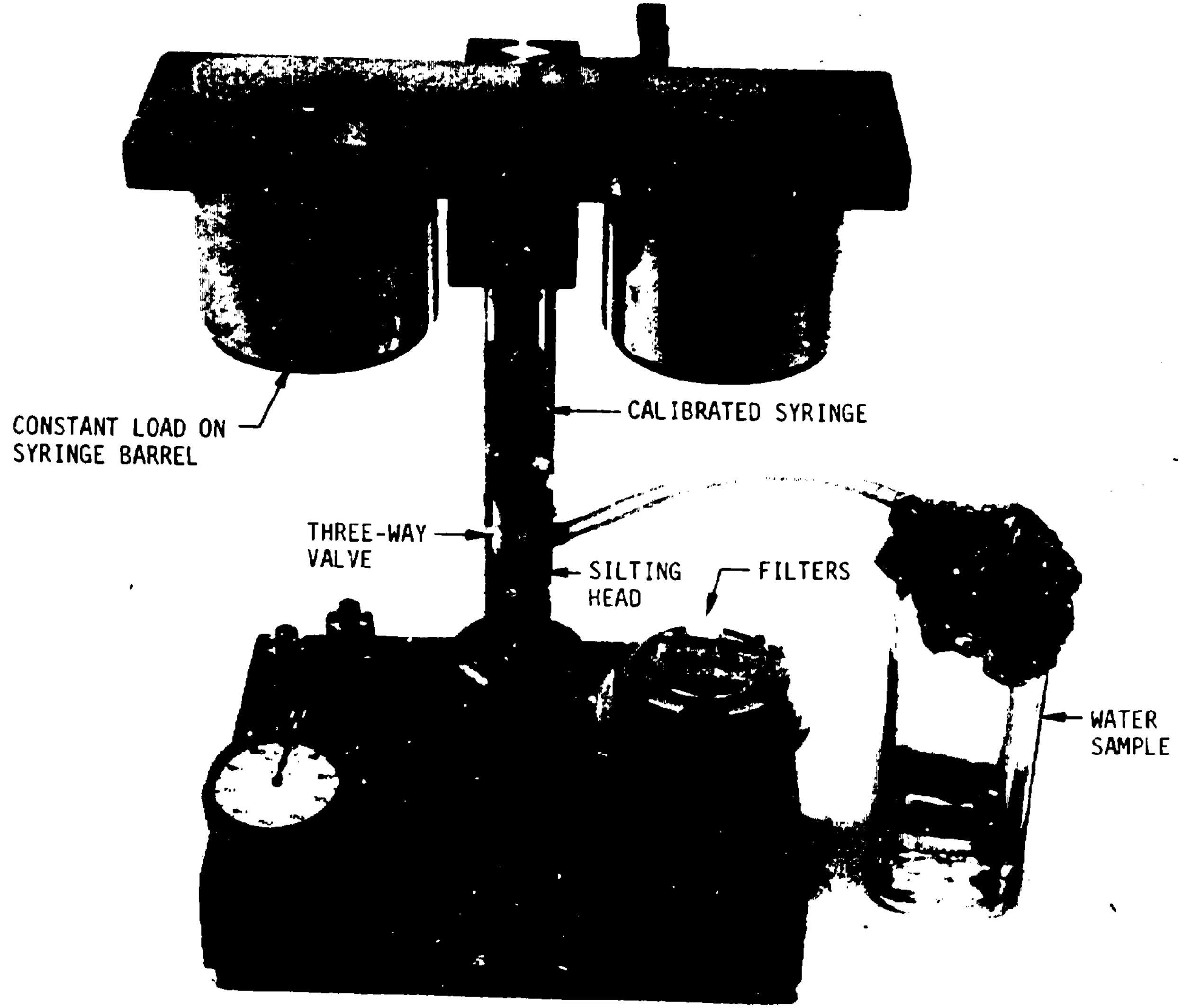

Figure B-2. Silting Index Apparatus

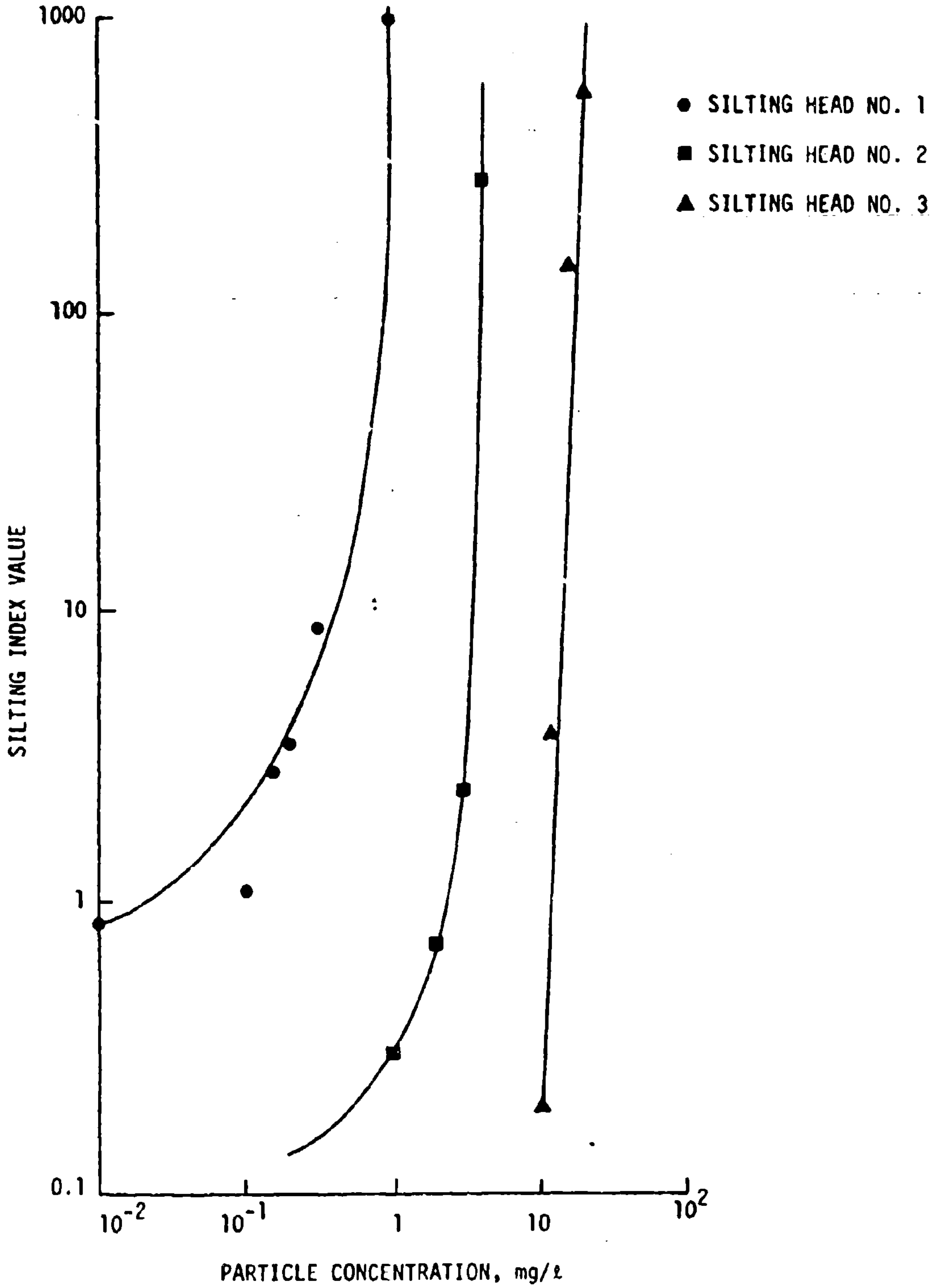

Figure B-3. Concentration Effect of 1.25 Micron Average Diameter Latex Particles on the Silting Index Values

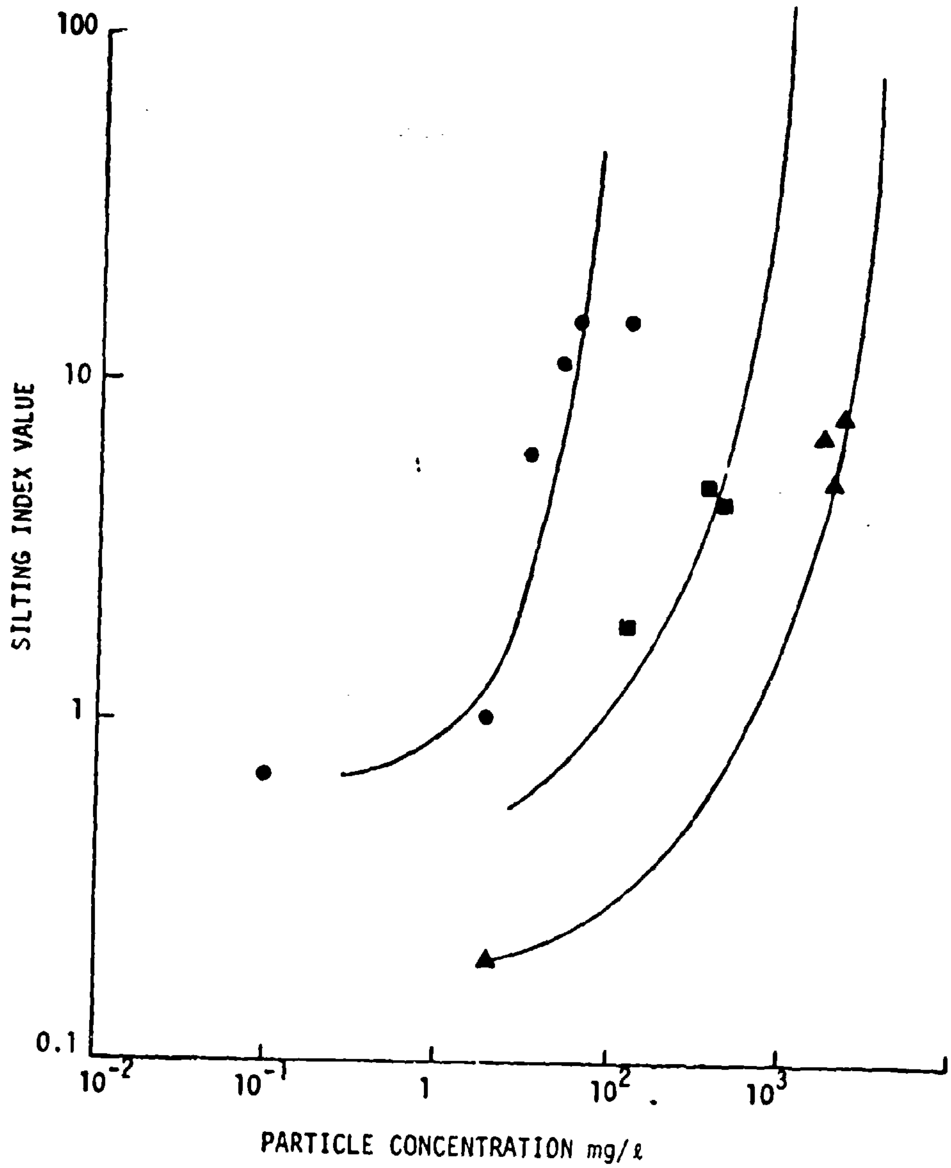

Figure B-4. Concentration Effect of 3-4 Micron Diameter Puff Ball Spores on the Silting Index Value densities of the particles are comparable. The significant item to note from the comparison of the data is that the Silting Index values are comparable at concentration levels that differ by at least one order of magnitude, the smaller particle producing the higher Silting Index value.